INSTITUTION OF PETROLEUM TECHNOLOGISTS

STANDARD METHODS of Testing Petroleum and Its Products

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PREFACE

THE importance of standardization has in recent years become increasingly apparent in scientific and technical progress. In the United States its development and application have already reached a high level, while in this country the British Engineering Standards Association has achieved satisfactory results in more than one direction with the co-operation of the industries concerned.

The suggestion that the Institution of Petroleum Technologists, as the premier society in this country dealing with petroleum technology, should concern itself with the standardization of methods of tests, was first mooted in a letter to the late Sir Boverton Redwood in 1917. At that time, although the Council was in favour of taking action, it was felt that war conditions precluded the securing of the attention necessary to the work. It was not until May, 1921, that the Standardization Committee was appointed.

The appropriate Government departments, the leading petroleum companies, and certain individual gentlemen of recognised authority were approached with the object of securing the co-operation of every branch of the industry, whether producing, merchanting, using, consulting or purely academic.

The complete Committee comprises the gentlemen whose names are given on p. vii., viii, and their occupation is stated in order that the diversity of interests and the degree of technological ability may be appreciated.

In order to expedite the work of the Committee, it was decided to divide its work into six classes and to appoint a Sub-Committee to deal with each class. The classes are as follows :—

(1) Naturally Occurring Bituminous Substances.
(2) Distillates up to Kerosenes.
(3) Kerosenes and Intermediates.
(4) Lubricants.
(5) Liquid Fuels.
(6) Asphaltum and Artificial Residues.

The names of the members of the Sub-Committees will be found on p. ix.

As it was felt that the main Committee would be too unwieldy to deal with the detail work of the Sub-Committees, an Executive Committee was appointed, consisting of the Chairman of each Sub-Committee and the Chairman of the main Committee. One of the functions of this Executive Committee is to secure definite decisions in cases where the recommendations of the Sub-Committees may not be unanimous. In the event of any test concerning more than one Sub-Committee, the respective Chairmen of the Sub-Committees meet, and if then there are any differences of opinion which they cannot reconcile, the matter is referred to the Executive Committee for decision. It is a remarkable fact that the Executive Committee has never had to exercise this function as regards any material difference of opinion.

The chief aim of the Committee has been to produce authoritative decisions as soon as possible in order to remove the difficulties under which petroleum technologists labour owing to divergences in methods and results. The spirit of the Committee throughout has been the reverse of parochial, and it has always given, and will continue to give, considerable weight to the valuable work achieved by the American Society for Testing Material and the American Bureau of Standards, and has, with the consent of these bodies, made use of their work and adopted many of their methods of test, and grateful acknowledgment is made to them for their willing assistance.

In the case of certain tests the Committee has not felt it possible to recommend departure from generally accepted methods, desirable as it is to secure uniformity with other countries—where such methods offer no grounds for technological objection—*e.g.*, the Abel closed flash-point apparatus is the one legally recognised throughout the British Empire ; then, again, the Redwood viscometers are so firmly established that any recommendation to adopt a different standard could only be justified by some serious shortcoming in the method.

The Committee decided to issue the approved methods as " Standard " and not as " Tentative " (the description used by our American confrères) because it was felt that the use of the word

"Tentative" would weaken the confidence of technologists, by giving an impression of indecision and indefiniteness. The use of the word "Standard," on the other hand, may possibly cause uneasiness to technologists as giving the impression of irrevocable permanency, but this is not the intention of the Committee, for it is intended that they shall continue their work and, from time to time, revise the tests as experience may demand and progress is made in methods and apparatus.

This first edition does not pretend to cover every test—in fact, is the first instalment; some of minor importance are still under consideration, but it was thought inadvisable to delay the publication of the work—which it is hoped will be of immediate and considerable use to petroleum technologists—in order to incorporate less important matter.

Not only have methods of testing been considered, but also the standardization of apparatus. The Committee's researches have revealed an unexpectedly grave variation in what have been generally regarded as standard instruments and, in order to secure certification of instruments, the Committee has arranged with the National Physical Laboratory that they will calibrate, test and mark hydrometers, thermometers, and, in specified cases, instruments as complying with the adopted standards. (Also p. iv.)

The British Engineering Standards Association was, in due course, approached with a view to avoiding duplication of work, and the Association decided that they would no longer concern themselves with methods of test of petroleum products, but would adopt, for the purposes of their specifications, whatever methods were standardized by the Institution.

The standardization of nomenclature has not yet been considered by the Committee, although it is their intention to review this matter at an early date. It is felt that nomenclature is not of prime importance to British Petroleum Technologists, and that when the time comes for considering it, it should be dealt with on very broad and international lines in the hope of bringing our nomenclature as far as possible into line with that used by the principal petroleum-producing countries.

The Committee have to acknowledge the valuable assistance

afforded by the principal firms of instrument manufacturers and by the British Lampblown Scientific Glassware Manufacturers' Association.

It might appear invidious to select any one member of the Committee for recognition of his work when all have worked so willingly and ably, but I am sure that every member would like me to record in this preface the debt which we all owe to Professor Brame, not only for his work as Chairman of a Sub-Committee, but for the remarkable amount of special work which he has carried out, including the editing of the whole of the Sub-Committees' work for publication.

In conclusion, it is hoped that the results so far obtained will be found of practical use to all those interested, and that, with a continuance of the spirit of co-operation, without which work on a subject such as Standardization cannot be effective, a comprehensive system may be attained and generally adopted, and which, while dealing satisfactorily with developments up to date, will, nevertheless, be of an elastic nature and will keep pace with the petroleum industry's progress.

ALEXANDER DUCKHAM.

VOLUMETRIC GLASSWARE.

When tests require the use of ordinary volumetric glassware, such as pipettes, burettes, etc., apparatus which has been tested at the National Physical Laboratory and passed Class A, or passed Class B, shall be used. In general the tests do not necessitate apparatus of a higher degree of accuracy than Class B.

CLASSES AND SERIAL DESIGNATION OF TESTS.

Petroleum products (including Crude Oils) have been divided in this Report into ten classes, to each of which reference letters have been given. The classes are as follows :—

No.	Class. Description.	Reference letter.	Pages.
I.	Gasoline	G. ..	5–13
II.	White Spirit (Turpentine substitute)	W.S. ..	14–15
III.	Kerosene	K. ..	16–23
IV.	Gas Oil (including special Vaporising Oils, Mineral Colza, and distillate oils for Heavy Oil Engines)	G.O. ..	24– 42
V.	Mineral Lubricating Oils	L.O. ..	43– 62
VI.	Transformer and Switch Oils	T.O. ..	63– 67
VII.	Fuel Oils (other than Intermediate Oils, Class IV.)	F.O. ..	68– 75
VIII.	Asphalts	A. ..	76– 91
IX.	Wax	W. ..	92– 95
X.	Crude Petroleum	C.P. ..	96–100

The Serial Designation has been arranged so that the same number is given to the same test throughout all classes, irrespective of the method of estimation employed. The serial number is preceded by the reference letter indicating the Class. Thus the estimation of sulphur always carries the number 4, so that G.4, K.4, GO.4 refer to this test for Gasoline, Kerosene and Gas Oil respectively

Where a test is specifically applicable to a particular product a high number has been allotted, thus the well-known "Doctor Test" for Gasoline has the I.P.T. Serial Designation G.33.

Dimensions of Apparatus.

The Committee fully recognise the desirability of adopting a consistent method of expressing units of measurement, preferably in the metric system, but this was found impossible, owing to the use of inch and fractions of an inch measurements in certain forms of apparatus which had to be adopted, and the customary use of

such fractions of an inch as thirty-seconds, sixty-fourths, etc. in workshops. Many of these work out to absurd decimals, or when converted into centimetres or millimetres, again give decimals of no practical utility.

A compromise had to be adopted, namely to accept the inch and decimals or fractions for metal parts, and centimetres or millimetres for glass and similar parts, and capacities for such vessels in cubic centimetres.

Again, both degrees Fahrenheit and Centigrade were forced on the Committee by existing conditions. Thus legal enactment for the Abel Flash Point apparatus prescribes the Fahrenheit scale, and the same scale is commonly employed in viscometry. For most methods such as distillations, the Centigrade system has been adopted.

List of Figures.

At a Council Meeting, held on May 30th, 1921, the following members, with power to co-opt additional members, were appointed as a "Standardization Committee": Dr. A. E. Dunstan, D.Sc., F.I.C., F.C.S. (Chief Research Chemist, Anglo-Persian Oil Co.), Dr. W. R. Ormandy, D.Sc., F.I.C., F.C.S., M.I.A.E. (Consulting and Analytical Chemist), Professor J. S. S. Brame, F.I.C., F.C.S. (Professor of Chemistry, R.N. College, Greenwich), Mr. James Kewley, M.A., F.I.C., F.C.S. (Chief Chemist, Shell Group), and Mr. Robert Redwood, F.C.S. (Consulting and Analytical Chemist). From these, Mr. Kewley was elected Chairman, but had to retire through pressure of other engagements. Mr. Alexander Duckham was co-opted and appointed Chairman, in which capacity he rendered valuable service.

The Government Departments were then approached and the following were appointed by their respective Departments :—

Admiralty.—Engineer Rear Admiral (then Eng. Captain) W. M. Whayman, C.B.E., R.N. (Deputy Engineer-in-Chief of the Navy).

War Office.—Mr. W. L. Baillie, F.I.C. (Chemical Inspection Department, Woolwich).

Air Ministry.—Dr. G. Rudorf, Ph.D. (Chemical Research Dept).

Government Laboratory.—Dr. J. J. Fox, O.B.E., D.Sc., F.I.C., F.C S. (Superintending Chemist).

National Physical Laboratory.—Mr. W. F. Higgins, M.Sc., A.R C.Sc. (Principal Assistant, Physics Department).

Petroleum Department, Board of Trade.—Mr. J. L. Jeffery, A.R.S.M., M.Inst.M.M. (Inspector of Drilling).

The leading oil companies were also approached and promised their support.

The following were from time to time co-opted as Members :—

Mr. Arthur A. Ashworth, M.A., A.M.Inst.C.E. (Messrs. Beeby, Thompson and Partners).

Mr. R. G. Batson, A.K.C., M.Inst.C.E., M.I.Mech.E. (National Physical Laboratory).

Mr. S. E. Bowrey, B.Sc. (Chemist, Alexander Duckham and Co.).

Mr. E. A. Evans, F.C.S. (Chemist, C. C. Wakefield and Co.).

Dr. F. H. Garner, Ph.D. (Chemist, Agwi Petroleum Corporation, Ltd.).

Mr. J. E. Hackford, B.Sc., F.I.C., F.C.S. (Consulting and Analytical Chemist).

Mr. Peter Kerr, M.A., B.Sc., A.I.C. (Chemist, Shell Group).

Mr. E. Lawson Lomax, M.Sc., F.C.S. (at that time with the Anglo-Persian Oil Co., Ltd.).

Mr. William Lee (Chemist, Silvertown Lubricants, Ltd.).

Mr. A. G. Marshall, M.A. (Research Chemist, Shell Group).

Mr. T. M. McKenzie, A.I.C., F.C.S. (Chemist, Anglo-Mexican Petroleum Co., Ltd.).

Mr. Arnold Philip, B.Sc., A.R.S.M., F.I.C., F.C.S. (Admiralty Chemist, Portsmouth).

Dr. P. E. Spielmann, Ph.D., B.Sc., F.I.C., A.R.C.Sc. (Highways Construction Co., Ltd.).

Dr. F. B. Thole, D.Sc., F.I.C., F.C.S. (Research Chemist, Anglo-Persian Oil Co., Ltd.).

Mr. H. T. Tizard, M.A. (Department of Industrial and Scientific Research).

Mr. W. J. Wilson, A.I.C. (Chemist, Burmah Oil Co., Ltd.).

Mr. W. A. Woodrow (Chemist, Anglo-American Oil Co., Ltd.).

The main committee has appointed the following six Sub-Committees :—

SUB-COMMITTEE 1.—*Naturally Occurring Bituminous Substances.*—James Kewley, M.A., F.I.C., F.C.S., A. E. Dunstan, D.Sc., F.I.C., F.C.S. (Chairmen); J. L. Jeffery, A.R.S.M., M.Inst.M.M., J.E. Hackford, B.Sc., F.I.C., F.C.S., W. J. Wilson, A.I.C., Robert Redwood, F.C.S., A. A. Ashworth, M.A., A.M.Inst.C.E., Peter Kerr, M.A., B.Sc., A.I.C., and F. B. Thole, D.Sc., F.I.C., F.C.S.

SUB-COMMITTEE 2.—*Distillates up to Kerosene.*—A. E. Dunstan, D.Sc., F.I.C., F.C.S. (Chairman), W. R. Ormandy, D.Sc., F.I.C., F.C.S., G. Rudorf, Ph.D., J. J. Fox, O.B.E., D.Sc., F.I.C., W. L. Baillie, F.I.C., S. E. Bowrey, B.Sc., W. J. Wilson, A.I.C., Peter Kerr, E. Lawson Lomax, M.Sc., F.C.S., H. T. Tizard, M.A., and A. G. Marshall, M.A.

SUB-COMMITTEE 3.—*Kerosene and Intermediates.*—Robert Redwood, F.C.S. (Chairman), Peter Kerr, M.A., B.Sc., A.I.C., W. J. Wilson, A.I.C., W. A. Woodrow, W. F. Higgins, M.Sc., A.R.C.Sc., and J. J. Fox, O.B.E., D.Sc., F.I.C.

SUB-COMMITTEE 4.—*Lubricants.*—F. B. Thole, D.Sc., F.I.C., F.C.S. (Chairman), S. E. Bowrey, B.Sc., W. F. Higgins, M.Sc., A.R.C.Sc., T. M. McKenzie, A.I.C., F.C.S., G. Rudorf, Ph.D., F. H. Garner, Ph.D., Eng. Captain W. M. Whayman, C.B.E., R.N., W. L. Baillie, F.I.C., Wm. Lee, and E. A. Evans, F.C.S.

SUB-COMMITTEE 5.—*Liquid Fuels.*—Professor J. S. S. Brame, F.I.C., F.C.S. (Chairman), Eng. Captain W. M. Whayman, C.B.E., R.N., F. H. Garner, Ph.D., W. J. Wilson, A.I.C., Arnold Philip, B.Sc., A.R.S.M., F.I.C., F.C.S., and W. A. Woodrow.

SUB-COMMITTEE 6.—*Asphaltum and Artificial Residues.*—J. E. Hackford, B.Sc., F.I.C., F.C.S. (Chairman), R. G. Batson, A.K.C., M.Inst.C.E., M.I.Mech.E., T. M. McKenzie, A.I.C., F.C.S., F. H. Garner, Ph.D., P. E. Spielmann, Ph.D., F.I.C., B.Sc., A.R.C.Sc.

NOTES ON SPECIFIC GRAVITY.

When specific gravity is required with accuracy in the fourth place of decimals and is determined by a weighing method (bottle, pyknometer, etc.) it is necessary to apply corrections for the buoyancy effect of the air.

When a specific gravity bottle or pyknometer is used

if Ww gms. = observed wt. in air of water content at 60° F.
and Ws gms. = ,, ,, sample contained in bottle (or pyknometer) at 60° F.

then the specific gravity S. $\frac{60^\circ F.}{60^\circ F.}$ is given *approximately* by

$$S^1 \frac{60^\circ F.}{60^\circ F.} = \frac{Ws}{Ww} \quad \ldots\ldots (1)$$

The approximate value given by the above formula may be corrected for the buoyancy effect of the air by means of the following relation :—

$$S \frac{60^\circ F.}{60^\circ F.} = S^1 \frac{60^\circ F.}{60^\circ F.} - 0{\cdot}0012 \left(S^1 \frac{60^\circ F.}{60^\circ F.} - 1\right) \quad \ldots\ldots (2)$$

where $S^1 \frac{60^\circ F.}{60^\circ F.}$ is the value obtained from (1) and $S. \frac{60^\circ F.}{60^\circ F.}$ is the corrected specific gravity.

The following table gives values of $\left\{ - 0{\cdot}0012 \left(S^1 \frac{60^\circ F.}{60^\circ F.} - \right) \right\}$:—

$S^1 \frac{60^\circ F.}{60^\circ F.}$	0·00	0·01	0·02	0·03	0·04	0·05	0·06	0·07	0·08	0·09
0·60	+5	+5	+5	+4	+4	+4	+4	+4	+4	+4
0·70	+4	+3	+3	+3	+3	+3	+3	+3	+3	+3
0·80	+2	+2	+2	+2	+2	+2	+2	+2	+1	+1
0·90	+1	+1	+1	+1	+1	+1	0	0	0	0
1·00	0	0	0	0	—1	—1	—1	—1	—1	—1
1·10	—1	—1	—1	—2	—2	—2	—2	—2	—2	—2

The values given in the table are expressed in units in the fourth decimal place, and may be used as corrections to be added when + and substracted when —.

If the specific gravity has to be determined at a temperature other than 60° F. then.

if Ww = observed wt. in air of water content at 60° F.
W^1s = ,, ,, sample contained in bottle at t° F.

the results are to be calculated as follows. Neglecting the expansion of the glass we have approximately

$$S^1 \frac{t}{60^\circ F.} = \frac{W^1s}{Ww} \quad \ldots\ldots (3)$$

or correcting for the buoyancy effect as before

$$S \frac{t}{60^\circ F.} = S^1 \frac{t}{60^\circ F.} - 0{\cdot}0012 \left(S^1 \frac{t}{60^\circ F.} - 1\right) \quad \ldots\ldots (4)$$

The values thus determined for $S \frac{t}{60^\circ F.}$ or $S^1 \frac{t}{60^\circ F.}$ are to be converted into specific gravities at 60° F.—*i.e.*, $S \frac{60^\circ F.}{60^\circ F.}$, by the addition ($t > 60^\circ F$) or subtraction ($t < 60^\circ F.$) of the constants given on p. 1.

METHODS OF TEST.

SPECIFIC GRAVITY.

Definition.—The term, specific gravity, is retained because its use is an established custom in Great Britain. The specific gravity of a substance is the ratio of the mass of any given volume of the substance to the mass of an equal volume of water. The temperature of the substance and of the water must be specified. The temperature 60° F. shall be employed in petroleum technology as the standard temperature for specific gravity determinations, and the results shall be expressed in terms of $S \frac{60° F.}{60° F.}$, signifying that the specific gravity in question is the ratio of the mass of any given volume of the substance to the mass of a quantity of water which, at 60° F., occupies a volume equal to that of the substance at 60° F.

Temperature.—Whenever the nature of the liquid permits, it shall be brought to 60° F. for the determination. With those liquids which necessitate the determination being carried out at a different temperature the result is to be corrected to 60° F. by the addition or subtraction of the following constants, per degree Fahrenheit :—

Products lighter than Kerosene :	Below 0·740	0·00048
	Above 0·740	0·00044
White Spirit		0·00042
Kerosene		0·00040
Gas Oils		0·00036
Diesel Engine Fuels		0·00035
Lubricating Oils		0·00034
Heavy Fuel Oils		0·00034
Melted Asphaltic bodies		0·00030

Apparatus.—The specific gravity bottle, or a suitable type of pyknometer, or a hydrometer to the general specification laid down as follows, shall be used.

All specific gravity bottles and pyknometers shall have their water value at 60° F. determined, and this value shall be used in all calculations.

For thick oils or tars to which the bottle or pyknometer is not suited the determination should be made in a 200 or 250 c.c. graduated flask, the water value of which has been determined as in the case of an ordinary specific gravity bottle. The oil or tar should be warmed before filling the bottle and the flask immersed in warm water up to the graduation mark for one hour

and air bubbles completely removed. The flask should then be cooled to 60° F., and the level of liquid adjusted to the mark. Alternatively, a temperature correction in accordance with the method and data above shall be made.

HYDROMETERS.

Hydrometers shall conform to the following general specifications :—

1. Hydrometers shall be made from glass, free from striæ and similar defects, and, in particular, the external surface of the stem shall be quite smooth. The glass shall be of a kind which sufficiently resists the action of chemicals, and possesses properties such as would render it suitable for use for thermometers —*i.e.*, it should have low thermal hysteresis and small secular change in volume.

2. Hydrometers shall be thoroughly annealed before the scale is fixed.

3. The main bulb shall be cylindrical, and the cross section of the stem circular.

4. A thermometer shall not be part of the instrument.

5A. When mercury is used for loading, it is to be contained in a bulb at the base of the hydrometer suitably sealed off with glass from the main bulb of the instrument.

5B. In special hydrometers for use at higher temperatures than 100° F. any material used for sealing off the bulb or for holding loading material, such as shot, shall not soften below 212° F.

(All hydrometers, after use at a higher temperature than 100° F., shall, as a matter of precaution, be allowed to drain and *cool* in a vertical position.)

6. The hydrometer shall be symmetrically constructed so that it floats with the stem vertical.

7. Paper of high quality shall be used for the scale conforming with the requirements that it shall not discolour at 212° F. with use, and that all divisions may be sharply and accurately marked on it.

8.—(*a*) The subdivisions of the scale shall be without evident irregularities.

(*b*) The graduation marks shall be made by fine straight lines, which lie in planes perpendicular to the axis of the hydrometer, so that the graduation marks are horizontal when the stem is vertical. The scale shall be straight and without twist and

fixed with a cement which will not soften at the highest temperature at which the hydrometer may be used.

(*c*) The scale of each hydrometer in Series A shall be graduated at each end for four divisions beyond its nominal range, for two divisions in Series B, and for one sub-division in Series C.

(*d*) Sufficient graduation marks shall be numbered to enable the exact reading at any point to be easily noted. At least every 10° graduation mark, *e.g.*, 650°, 660°, etc., shall be numbered. Abbreviated numbers, if used, must be confined to the central portion of the scale; the highest and lowest numbered graduation marks must be numbered in full. The numbers must not encroach on the space occupied by the shortest graduation marks, and must not touch or intersect any graduation mark.

(*e*) The length of the graduation marks must be varied so that the exact reading corresponding to any mark can be easily identified. The shortest graduation marks must be at least 2 mm. in length.

9. A fine horizontal line shall be etched on the stem of the hydrometer and this shall coincide with the horizontal part of a "pointing mark" which shall be ruled on the paper scale, about 5 mm. above the highest graduation. This "pointing mark" shall be a short, fine, line with a V at each end, thus >——<.

10. All hydrometers shall be graduated for reading correctly at the point of intersection of the level liquid surface with the stem. (When standard hydrometers are used with dark coloured liquids which do not permit of reading the intersection of the level liquid surface and stem, allowance must be made for the height of the meniscus round the stem, and the reading corresponding to the position of intersection of the level liquid surface and the stem must be estimated.)

The hydrometer must be adjusted so that the reading on the scale in a liquid whose temperature is 60° F. gives the specific gravity of the liquid at 60° F. relative to water at 60° F. The sp. gr. $\frac{60° \text{ F.}}{60° \text{ F.}}$ of water is to be taken as 1000, and the scale numbered accordingly.

11. The maker shall mark each hydrometer with

(*a*) S $\frac{60° \text{ F.}}{60° \text{ F.}}$ at 60° F.

(*b*) "I.P.T." and an identification number.

(*c*) Vendor's or maker's name or mark.

(*d*) "Max. Temp. 212° F." or "Max. Temp. 100° F.," according to whether the cementing material for fixing the loading is or is not suitable for the hydrometer to be used above 100° F.

Distinguishing Marks on Standard Instruments.

12. No hydrometer shall be regarded as "standard" unless it has been tested by the National Physical Laboratory for accuracy and dimensions, and certified by the National Physical Laboratory monogram being etched upon the instrument.

Hydrometer Series.

The following three series of hydrometers covering a range in each series of 650 to 1100 specific gravity, in nine separate instruments, each having a range of 50 degrees—*i.e.*, covering a change in specific gravity of 0·05, have been adopted :—

	Series A.	Series B.	Series C.
Length overall not more than ..	340 mm.	250 mm.	175 mm.
Diameter of bulb, nowhere to exceed	40 mm.	30 mm.	25 mm.
Graduations, representing equal increments of density	100 divisions	50 divisions	25 divisions
Length of nominal 50° portion of scale { not less than	155 mm.	75 mm.	50 mm.
Length of nominal 50° portion of scale { not more than	175 mm.	90 mm.	60 mm.
Highest graduation mark from top of stem—not less than	25 mm.	20 mm.	20 mm.
The length of plain cylindrical stem below the lowest graduation mark shall be not less than ..	5 mm.	5 mm.	5 mm.

The external diameter of the stem of the individual hydrometers shall be not less than the lower figure or more than the higher figure given below :—

Range.	A. mm.	B. mm.	C. mm.
650—700 ..	7·5 : 8·0 ..	8·0 : 8·4 ..	6 9 : 7·2
700—750 ..	7·2 : 7·7 ..	7·7 : 8·1 .	6·7 : 7·0
750—800 ..	6·9 : 7·4 ..	7·5 : 7·9 ..	6·5 : 6·8
800—850 ..	6·7 : 7·2 ..	7·2 : 7·6 ..	6·3 : 6·6
850—900 ..	6·5 : 7·0 ..	7·0 : 7·4 ..	6·1 : 6·4
900—950 ..	6·3 : 6·8 ..	6 8 : 7·2 ..	5·9 : 6·2
950—1000 ..	6·1 : 6·6 ..	6·6 : 7·0 ..	5·7 : 6·0
1000—1050 ..	6·0 : 6·5 ..	6·4 : 6·8 ..	5·6 : 5·9
1050—1100 ..	5·8 : 6·3 ..	6·2 : 6·6 ..	5·5 : 5 8

The tolerance of accuracy at any point for hydrometers in the three series shall be :—

A.	B.	C.
± 0·4° Sp. gr. ..	± 1·0° Sp. gr. ..	± 1·5° Sp. gr.

CLASS I.—GASOLINE

SPECIFIC GRAVITY.

I.P.T. Serial Designation—G.1.

The apparatus and method shall be as laid down in the special directions for Specific Gravity (p. 1).

COLOUR.

I.P.T. Serial Designation—G.2.

APPARATUS.

The Lovibond Tintometer shall be employed, as laid down for Kerosene—K 2 (p. 16).

DISTILLATION.

I.P.T. Serial Designation—G.3.
A.S.T.M. Serial Designation—D86—23 T. (modified)

APPARATUS.

DISTILLATION.—*Apparatus.* The Standard Apparatus shall comply with the following specification.

Flask.—The standard 100 c.c. Engler flask is shown in Fig. 1, the dimensions and allowable tolerance being as follows :—

	Centimetres.	Tolerances in centimetres.
Diameter of bulb, outside	6·5	0·2
„ „ neck, inside	1·6	0 1
Length of neck	15·0	0·4
„ „ vapour tube	10·0	0 3
Diameter of vapour tube—		
Outside	0·6	0 05
Inside	0·4	0 05
Thickness of vapour tube wall	0·1	0·05

The position of the vapour tube shall be 9 cm. (3·55 in.) ± 3 mm. above the surface of the liquid when the flask contains its charge

of 100 c.c. The tube is approximately in the middle of the neck and set at an angle of 75 deg. (tolerance ± 3 deg.) with the vertical.

Condenser.—The condenser shall consist of a uniform glass 56 cm. (22 in.) in length, and 1·25 cm. (±2 mm.) ($\frac{1}{2}$ in.) internal diameter. The lower end of the tube shall be attached to an

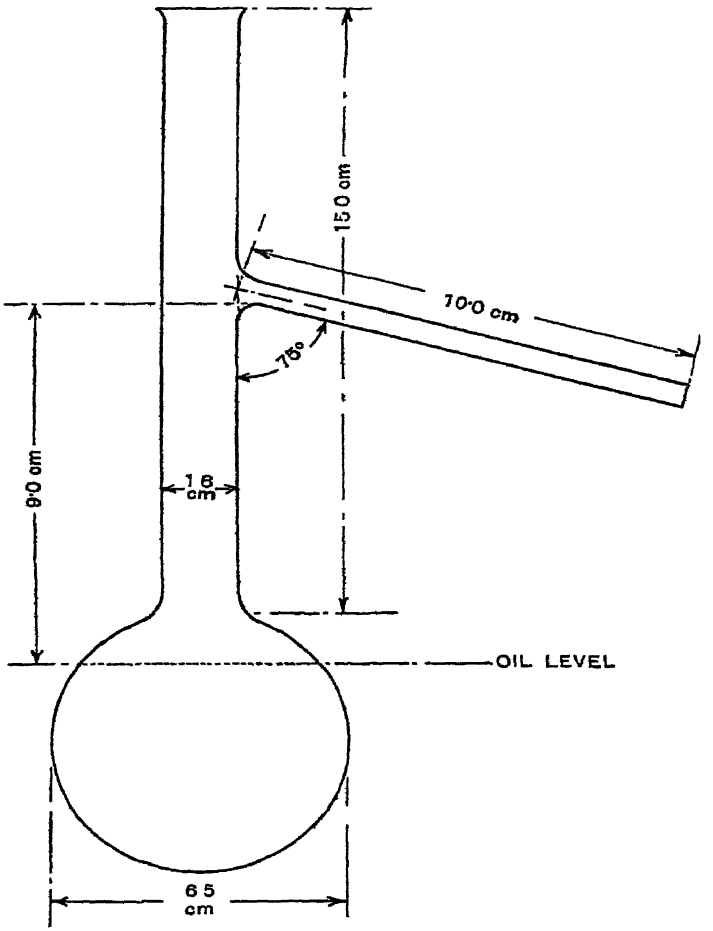

FIG. 1.
STANDARD DISTILLATION FLASK.

adapter or bent downwards so that its lowest point may touch the side of the receiving vessel. The condenser shall be cooled by an outer water jacket, 38-40 cm. (15 in.) in length, through which tap water flows.

Shield and Support.—The flask shall stand in a vertical position on a sheet of asbestos 6 in. square, in the centre of which is a circular hole 1¼ in. in diameter. The flask, support and source of heat shall be surrounded by an efficient screen to eliminate the disturbing effects of draughts.

Thermometer Specification.

Type.—Mercury in glass. Solid stem. Nitrogen filled.

Stem.—Lead glass or other suitable glass. Enamel back. Diameter, 5·5 to 7 mm.

Bulb.—Cylindrical. Powell's normal glass, Jena 16^{111}, or other approved glass. Length, 10 to 15 mm. Diameter, less than stem.

Range.—0° to 300° C.

Immersion.—Total.

Dimensions.—Overall length, approximately 380 mm. The distance from the bottom of the bulb to the 35° mark shall not be less than 110 mm. or more than 135 mm. The total length of the graduated portion shall be not less than 210 mm. or more than 250 mm.

Expansion Chamber.—Required.

Graduation.—The scale divided to 1° C. divisions. Every 5° and 10° shall be indicated by longer lines.

Figuring.—The scale shall be figured at each 10°

Marking.—"I.P.T. Low Distillation." Identification number. Maker's (or vendor's) name or trade mark.

Each thermometer shall be tested at the National Physical Laboratory where satisfactory thermometers will be marked with the N.P.L. monogram and year of test, and a certificate of corrections issued.

Graduated Receiver.—The graduate shall be of the cylindrical type, of uniform diameter, with a pressed or moulded base and a lipped top. The cylinder shall be graduated to contain 100 c.c , and the graduated portion shall be not less than 18 cm. (7 in.), and not more than 20 cm. (8 in) long. It shall be graduated in single cubic centimetres, and each fifth mark shall be distinguished by a longer line. It shall be numbered from the bottom up at intervals of 10 c.c. The distance from the 100 c.c. mark to the rim shall be not less than 3 cm. (1¼ in.), nor more than 4·5 cm. (1¾ in.). The receiver shall comply with the accuracy requirements of the National Physical Laboratory for Class B graduated glassware.

METHOD.

The flask shall be clean and dry. The condenser shall be rinsed with a little of the liquid under test and allowed to drain for three minutes. 100 c.c. of the liquid to be tested shall be measured in the graduated jar and transferred to the flask, special care being taken that the side arm of the flask is not wetted. The graduated jar shall be used without drying, as receiver.

The thermometer, the bulb of which shall be covered with a fresh uniform layer of cotton wool, 5 mgs. in weight, shall be inserted in the flask so that the lower end of the capillary is level with the lower edge of the side arm at its point of attachment to the flask.

The flask shall be attached to the condenser so that the end of the side arm is central in the condenser tube and projects 2·5 cm. beyond the cork. The end of the condenser or adapter shall touch the side of the receiver the open mouth of which shall be covered with wet filter paper.

The preliminary period between starting the heating and the appearance of first drop shall be a minimum of five minutes, and shall not exceed ten minutes.

The flame shall be set at a size to give a rate of distillation of 4–5 c.c. per minute (approximately 2 drops per second) and suitably increased up to 90 per cent. off; then left at this size till the final boiling point is reached. The initial boiling point shall be the temperature recorded when the first drop of liquid falls from the side arm of the flask, and the final boiling point the temperature recorded when the bottom of the flask becomes dry.

The condenser shall be allowed to drain for three minutes before measurement of total distillate.

Measurements to be recorded.

I.B.P. and F.B.P. (Recommended tolerance of 5° C.).

Distillate at 50° C.	Distillate at 150° C.
,, ,, 75° C.	,, ,, 175° C.
,, ,, 100° C.	,, ,, 200° C.
,, ,, 125° C.	,, ,, 225° C.

Total distillate. Residue in flask. Loss. Barometer pressure.

No corrections for barometric pressure, nor for exposed thermometer stem shall be applied.

TOTAL SULPHUR.

I.P.T. Serial Designation—G. 4.
A.S.T.M. Serial Designation D 90—21 T. (with modification).

APPARATUS (FIG. 2).

The apparatus employed shall consist of :—

A. A glass bottle, squat shape, 15 c.c. capacity, fitted with cork, and transparent silica tube, 2·5 cm. by 4–5 mm., holding a length of lead free glass wool which acts as a wick. This material is superior to cotton wick on account of its non-adsorbent properties.

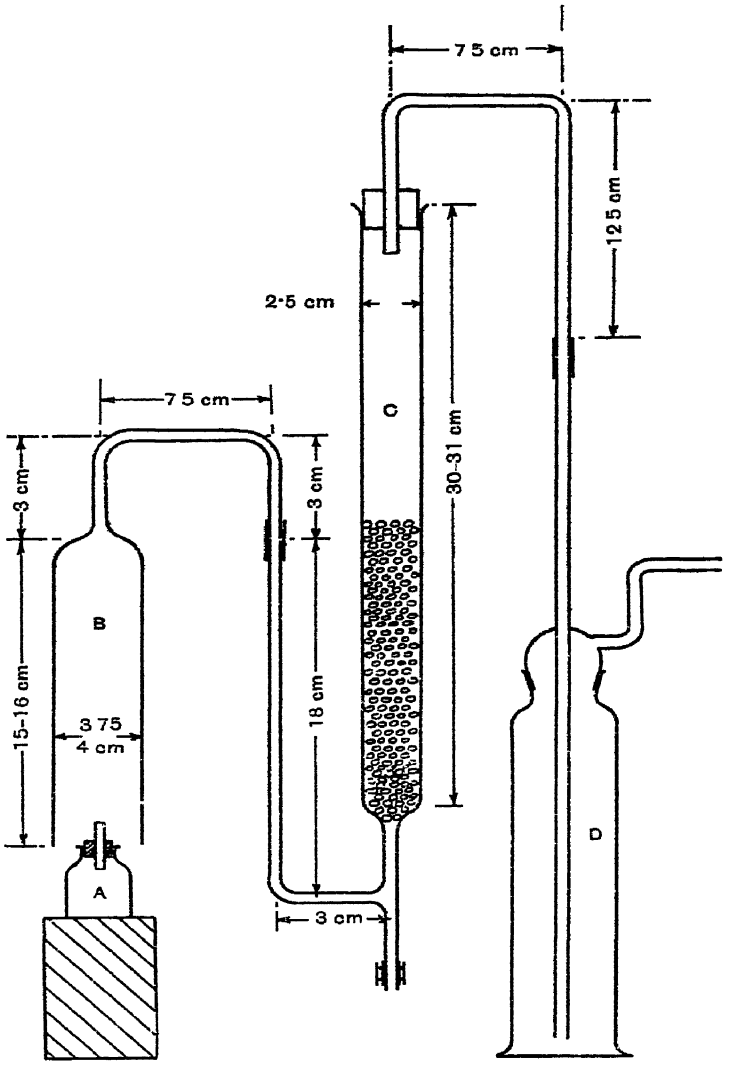

FIG. 2.

SULPHUR APPARATUS.

B. A glass chimney, 15–16 cm. long, 3·75–4 cm. diameter, with tube bent twice at right angles, as in Fig. 2.

C. A glass absorbing vessel half filled with 4–5 mm. glass balls. The tube shall be 30 cm. long, 2·5 cm. diameter, with a right-angle tube at the bottom and extension for draining contents.

D. A 100 cc. Drechsel wash bottle which acts as a trap for any spray carried over from C.

E. Filter pump or other means for continuous suction with rubber tubing necessary for connection to D.

REAGENTS.

Sodium carbonate solution, 10 per cent.

Sodium peroxide, pure—solid.

Hydrochloric acid.—Concentrated.

Barium chloride.—100 grams per litre.

Barium nitrate.—Saturated solution.

Ammonium nitrate solution, 10 per cent.

METHOD.

For gasoline, 5 c.c. of the sample (the specific gravity of which is known) shall be pipetted into the lamp. The vessel C shall be charged with 20 c.c. of 10 per cent. sodium carbonate solution, and D with 20 c.c. of water and 1 c.c. of 10 per cent. sodium carbonate solution. Steady suction shall be applied to the apparatus so that the burning lamp gives a flame $\frac{1}{2}$–$\frac{3}{4}$ in. in height, and just free from smoking, which must be avoided.

When burned to dryness the lamp shall be rinsed out twice with 2 c.c. of amyl acetate, burning to dryness in each case. The lamp and wick should be now free from all traces of the oil and ready for another test.

Under these conditions, the burning of gasoline should be completed in 2 hours, and of the heavier distillates in 3 hours. The contents of C and D and all connections shall be thoroughly washed out into a beaker, keeping the bulk of liquid as small as possible, and not exceeding 200 c.c. To the combined washings shall be added about 0·5 grams of sodium peroxide and the liquid heated to boiling, made slightly acid with hydrochloric acid and 5 c.c. of a boiling solution of barium nitrate or of barium chloride added.

The solution shall be kept on a hot plate or top of a steam bath for not less than 4 hours. It is recommended that it shall stand all night, with a suitable cover to prevent loss by evaporation.

The paper and precipitate shall be washed with water until free from all trace of chlorides, and finally with a solution of ammonium nitrate (10 per cent.) and dried. This treatment assists combustion of the paper and prevents reduction of the barium sulphate. The paper shall be burnt off and the precipitate and paper ash ignited and weighed in the usual manner.

A blank experiment shall be carried out under identical conditions in order that allowance may be made for traces of sulphates in the reagents used. Determinations shall be carried out in a room the air of which is not liable to contamination with sulphurous products.

AROMATIC CONTENT.

I.P.T. Serial Designation—G.30.

Aromatic Content.—The critical solution temperature (C.S.T.) of the spirit in freshly distilled pure aniline is determined before and after removal of the aromatic hydrocarbons by means of 98–100 per cent. sulphuric acid. It is essential that the concentration of the acid shall lie between these limits.

APPARATUS.

For the determination of the C.S.T., the following apparatus shall be employed :—

(1) A test tube, 25 mm. diameter and about 150 mm. long, provided with a glass or stiff wire stirrer and a thermometer, fixed in a wider tube as air jacket and heated in a beaker of water.

(2) A thermometer graduated to cover a range from 0° C. to 100° C., and with each degree interval sub-divided into 1/5°.

METHOD.

5 c.c. of the spirit under test shall be placed in the tube, mixed with 3 c.c. of freshly distilled aniline run in from a 10 c.c. burette, and the temperature of miscibility taken. Repeat experiments shall be made after successive additions of 0·5 c.c. aniline till the maximum temperature at which complete miscibility just occurs is found ; this maximum is the critical solution temperature.

The removal of the aromatics shall be done as follows : 20 c.c. of the spirit are continuously shaken with 70 c.c. of 98–100 per cent. sulphuric acid in a stoppered tap funnel for 15 minutes ; the spent acid run off and the spirit continuously shaken for a further 15 minutes with a further 20 c.c. of the acid. The residual spirit shall then be washed with water and caustic soda and dried with calcium chloride.

The percentage by weight of aromatic hydrocarbons shall be calculated by multiplying the increase of C.S.T. by 1·19.

To calculate results as percentage by volume the following formula shall be used :—

$$Y = 100 - (100 - X)\frac{S_1}{S_2}$$

where X = Percentage of aromatics by weight.
Y = " " " " volume.
S_1 = Specific gravity of original sample.
S_2 = " " after removal of aromatics.

The method is not applicable to gasoline containing appreciable amounts of olefine hydrocarbons.

The factor 1·19 being the mean of the factors for benzene, toluene and xylene is only a first approximation. The error arising is, however, small and of the same order as the experimental error. If the aromatic content exceeds 20 per cent. the spirit shall be diluted with a suitable quantity of the same spirit previously freed from aromatic hydrocarbons before examination, as the factor 1·19 does not hold for a concentration exceeding 20 per cent.

For the separate determination of benzene, toluene and xylene, the following procedure shall be employed.

500 c.cs. of the spirit are carefully fractionally distilled at a speed of 1 drop per second, using an efficient column ; three cuts are made, up to 95°, 95° to 122°, and 122° to 150° C. The volumes of the fractions are measured and the C.S.T. of each determined before and after sulphonation, according to the method described above.

The appropriate multiplying factors to convert the increase of C.S.T. to percentage by weight of aromatic hydrocarbons are :—

Benzene fraction	up to 95° C.	.. ..	1·15
Toluene ,,	95° to 122° C.	.. . .	1·20
Xylene ,,	122° to 150° C.	.. ..	1·23

These cutting points are not accurate when one aromatic hydrocarbon greatly predominates over the others.

The percentage by volume shall be calculated by the same method as that laid down for " total " aromatics by volume as given above.

" DOCTOR " TEST.

I.P.T. Serial Designation—G. 33.

REAGENTS.

(1) *Sodium Plumbite* (*Doctor solution*).—The solution shall be prepared as follows. Dissolve approximately 125 grams of sodium hydroxide (NaOH) in a litre of distilled water. Add 60 grams. of litharge (PbO) passing a 100-mesh sieve, boil for half an hour or shake vigorously for 15 minutes or let stand with occasional shaking for at least a day. Allow to settle and decant or siphon off the clear liquid. Filtration through a mat of asbestos may be employed if the solution does not clear. The solution shall be kept in a tightly corked bottle and shall be refiltered before use if not clear.

(2) *Sulphur*.—Pure, dry flowers of sulphur.

METHOD.

10 c.c. of the sample shall be shaken vigorously in a test tube for 15 seconds with 5 c.c. of sodium plumbite solution. A small pinch of sulphur shall be added, and the whole shaken for a further 15 seconds and allowed to settle. The quantity of sulphur used shall be such that practically all of it floats on the interface between the sample and the sodium plumbite solution.

INTERPRETATION OF RESULTS.

If the sample is discoloured, or if the yellow colour of the sulphur film is noticeably masked, the test shall be reported as "positive" and the sample condemned as "sour." If the sample remains unchanged in colour, and if the sulphur film is bright yellow, or only slightly discoloured with gray or flecked with black, the test shall be reported "negative," and the sample considered "sweet."

CORROSION TEST.

(COPPER DISH.)

I.P.T. Serial Designation—G. 32.

APPARATUS.

A freshly polished hemispherical dish of spun copper, approximately 3·5 inches in diameter.

METHOD.

100 c.c. of the gasoline to be examined shall be placed in the dish and the dish placed in an opening of an actively-boiling steam-bath, so that the steam comes in contact with the outer surface of the dish up to the level of the gasoline. The dish shall remain on the steam-bath until all volatile matter has disappeared.*

INTERPRETATION OF RESULTS.

If the gasoline contains dissolved elementary sulphur or corrosive sulphur compounds, the bottom of the dish will be coloured gray or black.

If the gasoline contains undesirable gum-forming constituents there will be a weighable amount of gum deposited on the dish. Acid residues will show as gum in this test.

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* Special precautions must be taken to avoid fire. An electrically-heated bath is to be preferred.

CLASS II.—WHITE SPIRIT

(TURPENTINE SUBSTITUTE)

SPECIFIC GRAVITY.

Serial Designation—W.S. 1.

The apparatus and method shall be as laid down in the special directions for specific gravity (p. 1).

COLOUR.

I.P.T. Serial Designation—W.S. 2.

The Lovibond Tintometer shall be used, as laid down under G. 2 (p. 16).

DISTILLATION.

I.P.T. Serial Designation—W.S. 3.

The apparatus and procedure shall be as laid down under G. 3 (p. 5).

FREE ACIDITY.

I.P.T. Serial Designation—W.S. 5.

The method shall be as laid down under L.O. 5 (p. 43).

FLASH POINT.

I.P.T. Serial Designation—W.S. 7.

The Abel apparatus shall be employed according to the procedure laid down under K. 7 (p. 18).

NON-VOLATILE MATTER.

I.P.T. Serial Designation—W.S. 42.

50 c.c. of the material shall be evaporated in a shallow dish (approximately 10 cm. in diameter, and 4 cm. deep) on a boiling water bath for four hours.* The residue shall be weighed and the result reported as percentage by weight. The presence of inorganic matter, if any, shall also be reported.

OILY MATTER.

I.P.T. Serial Designation—W.S. 43.

To detect the presence of oily matter 0·2 c.c. of the spirit shall be allowed to fall on the centre of a piece of white ashless filter paper 11 cm. in diameter. The paper shall then be suspended vertically from a glass hook and freely exposed to the air for one hour at a temperature of approximately 60° F. and any oily residue noted.

* Proper precautions should be taken against fire. (See G. 32.)

CLASS III.—KEROSENE

SPECIFIC GRAVITY.

I.P.T. Serial Designation—K. 1.

The apparatus and method shall be as laid down in the special directions for specific gravity (p. 1).

COLOUR.

I.P.T. Serial Designation—K. 2.

APPARATUS.

The Lovibond Tintometer, fitted with an 18-in. cell to contain the oil to be tested, shall be employed.

The instrument is provided with four special standard glasses : Water White (1·0), Superfine White (2·0), Prime White (3·0), and Standard White (4·0), and with intermediate glasses.

METHOD.

Illumination.—It is recommended that whenever practicable an " Artificial Daylight " type of electric lamp shall be used for illumination.

Where daylight has to be employed the apparatus shall be placed facing a north light. Direct sunlight must be avoided.

Other light must be excluded from the instrument.

The instrument shall be adjusted so that even illumination of both sides is obtained.

The liquid to be examined shall be placed in the 18-in. cell, sufficient of the sample being used to cover the whole rectangular space of the cell when viewed through the eyepiece.

The standard and intermediate glasses shall be placed in turn in the slots on the other side of the instrument until the glass showing the colour most nearly approximating to that of the sample is ascertained.

EXPRESSION OF RESULTS.

If a sample is found to be darker than any one of the standard glasses the colour shall be described by the next standard, the inter-

mediate glasses being used to determine the position of the colour between the two standards.

For example, if the colour is midway between Superfine White (2·0) and Prime White (3·0) it is to be expressed as Prime White (2·5).

DISTILLATION TEST.

I.P.T. Serial Designation—K. 3.

A.S.T.M. Serial Designation—D 86—23 T (modified).

The apparatus and method shall be as described under G.3 (p. 5).

The asbestos card on which the flask rests shall have a hole 2 inches in diameter in this case.

THERMOMETER SPECIFICATION.

Type.—Mercury in glass. Solid stem. Nitrogen filled.

Stem.—Lead glass or other suitable glass. Enamel back. Diameter, 5·5 to 7 mm.

Bulb.—Cylindrical. Powell's normal glass, Jena 16^{111}, or other approved glass. Length, 10 to 15 mm. Diameter, less than stem.

Range.—0° to 400° C.

Immersion.—Total.

Dimensions.—Overall length, approximately 400 mm. The distance from the bottom of the bulb to the 0° C. mark not less than 25 mm. or more than 30 mm. The total length of the graduated portion (0/400° C.) shall be not less than 325 mm. or more than 350 mm.

Expansion Chamber.—Required.

Graduation.—The scale divided to 1° C. divisions. Every 5° and 10° shall be indicated by longer lines.

Figuring.—The scale shall be figured at every 10° C.

Marking.—" I.P.T. High Distillation." Identification number. Maker's (or vendor's) name or trade mark.

Each thermometer shall be tested at the National Physical Laboratory, where satisfactory thermometers will be marked with the N.P.L. monogram and year of test, and a certificate of corrections issued.

TOTAL SULPHUR.

I.P.T. Serial Designation—K. 4.

A.S.T.M. Serial Designation—D. 90—21 T. (with modifications).

The apparatus and method shall be as laid down under G.4 (p. 8).

FREE ACIDITY.

I.P.T. Serial Designation—K. 5.

The method shall be as laid down under L.O. 5 (p. 43).

CALORIFIC VALUE.

I.P.T. Serial Designation—K. 6

The apparatus and method (bomb calorimeter) shall be as laid down under G.O. 6 (p. 26).

FLASH POINT.

I.P.T. Serial Designation—K. 7.

The flash point shall be determined by the Abel apparatus as defined by the Petroleum Acts, provided that the flash point is not above 120° F., and by the Pensky-Martens apparatus above that temperature.

The Abel apparatus and thermometers shall be certified and marked by the Board of Trade as complying with the requirements of the Petroleum Acts. The Pensky-Martens apparatus shall be similarly certified by the National Physical Laboratory.

APPARATUS (ABEL).

The oil-cup (Fig. 3) consists of a cylindrical vessel 2 in. diameter, 2·2 in. height (internal), with outward projecting rim 0·5 in. wide. 0·375 in. from the top, and 1·875 in. from the bottom of the cup, It is made of gunmetal or brass (17 B.W.G.) tinned inside. A bracket (B), consisting of a short, stout piece of wire bent upwards and terminating in a point, is fixed to the inside of the cup to serve as a gauge. The distance of the point from the bottom of the cup is 1·5 in. The cup is provided with a close-fitting overlapping cover made of brass (22 B.W.G.), which carries the thermometer (C) and test-lamp (D). The latter is suspended from two supports from the side by means of trunnions upon which it may be made to oscillate ; it is provided with a spout, the mouth of which is one-

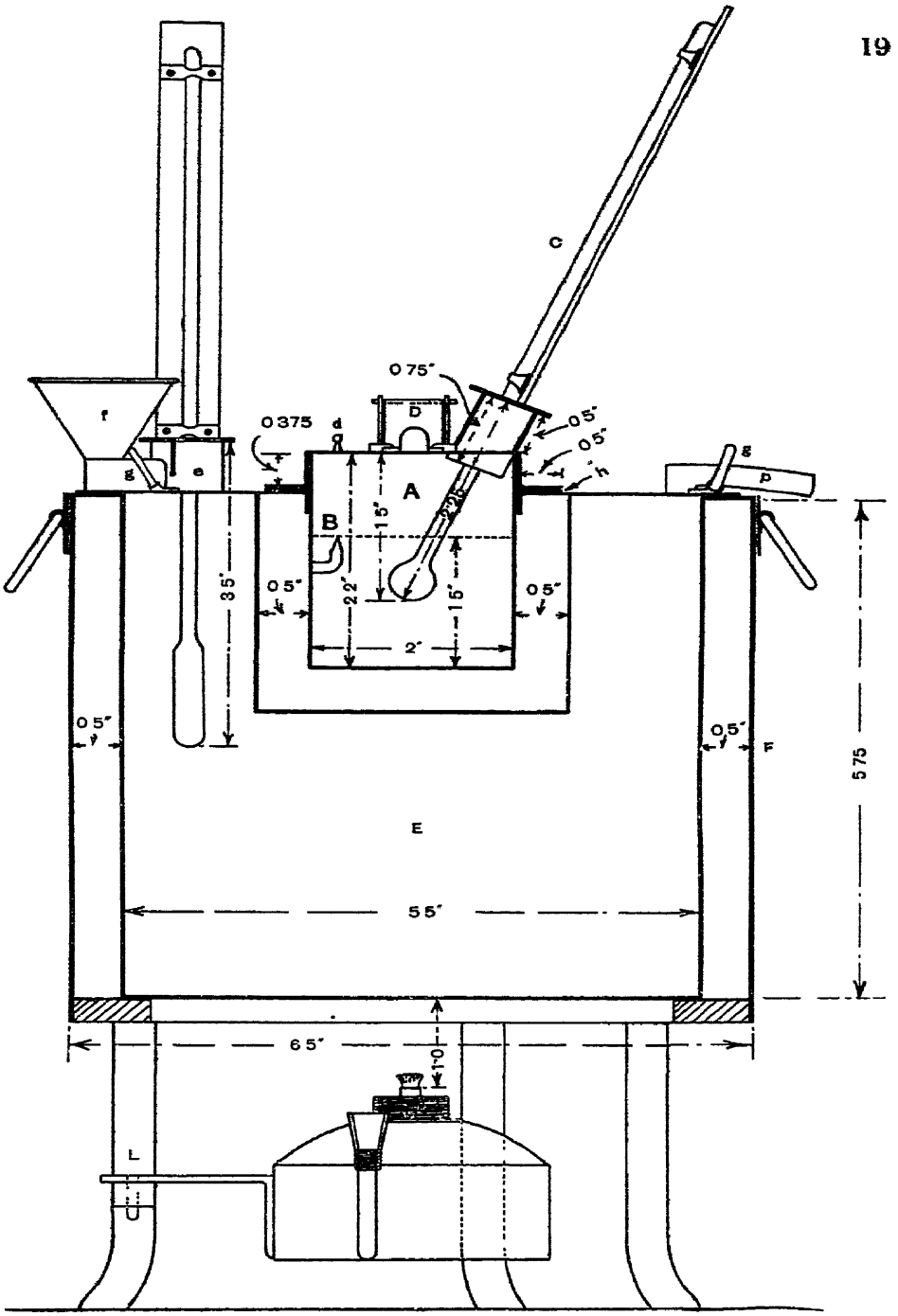


FIG. 3. ABEL FLASH-POINT APPARATUS.

sixteenth of an inch in diameter. The socket which is to hold the thermometer is fixed at such an angle and its length is so adjusted that the bulb of the thermometer when inserted to its full depth shall be 1·5 in. below the centre of the lid.

The cover, Figs. 4 and 5, is provided with three square holes, one in the centre, 0·5 in. by 0·4 in., and two smaller ones, 0·3 in. by 0·2 in., close to the sides and opposite each other (Fig. 5). These three holes may be closed and uncovered by means of a slide moving in grooves, and having perforations corresponding to those on the lid.

In moving the slide so as to uncover the holes, the oscillating lamp is caught by a pin fixed in the slide, and tilted in such a

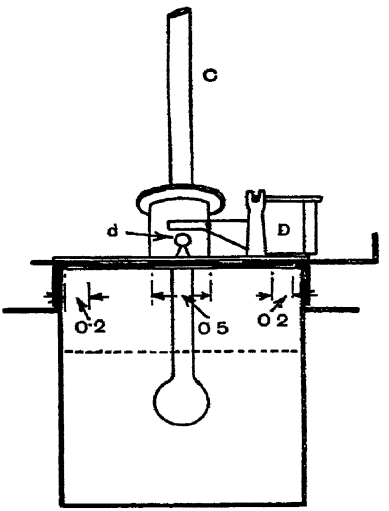


FIG. 4.

CUP OF ABEL FLASH-POINT APPARATUS.

way as to bring the end of the spout just below the surface of the lid. Upon the slide being pushed back so as to cover the holes, the lamp returns to its original position.

Upon the cover, in front of and in line with the mouth of the lamp, is fixed a white bead, Fig. 3 (*d*), the dimensions of which represent the size of the test-flame to be used.

The bath or heating vessel (E, Fig. 3) consists of two flat-bottomed copper cylinders (24 B.W.G.), an inner one of 3 in. diameter and 2·5 in. height, and an outer one of 5·5 in. diameter and 5·75 in. height; they are soldered to a circular copper plate (20 B.W.G.) perforated in the centre, which forms the top of the bath, in such a manner as to enclose

the space between the two cylinders, but leaving access to the inner cylinder. The top of the bath projects both outwards and inwards about 0·375 in.; that is, its diameter is about 0·750 in. greater than that of the body of the bath, while the diameter of the circular opening in the centre is about the same amount less than that of the inner copper cylinder. To the inner projection of the top is fastened, by six small screws, a flat ring of ebonite, the screws being sunk below the surface of the ebonite, to avoid metallic contact between the bath and the oil-cup, Fig. 3 (*h*). The exact distance between the sides and bottom of the bath and of the oil-cup is 0·5 in. A split socket, Fig. 3 (*e*), similar to that on the cover of the oil-cup, but set at a right angle, allows a thermometer to be inserted into the space between the two cylinders. The bath is further provided with a funnel, Fig. 3 (*f*), an overflow pipe, Fig. 3 (*p*), and two loop handles, Fig. 3 (*g*).

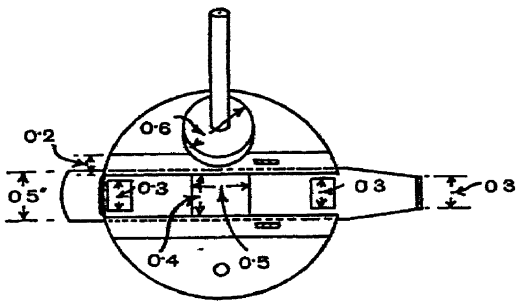


FIG. 5.
COVER ABEL FLASH-POINT APPARATUS.

The bath rests upon a cast-iron tripod stand, to the ring of which is attached a copper cylinder or jacket, Fig. 3 (F) (24 B.W.G.) flanged at the top, and of such dimensions that the bath, while firmly resting on the iron ring, just touches with its projecting top the inward-turned flange. The diameter of this outer jacket is 6·5 in. One of the three legs of the stand serves as support for the spirit lamp attached to it by means of a small swing bracket, Fig. 3 (L). The distance of the wick-holder from the bottom of the bath is 1 in.

Two thermometers are provided with the apparatus, the one for ascertaining the temperature of the bath, the other for determining the flashing point. The thermometer for ascertaining the temperature of the water has a long bulb and a space at the top. Its range is from about 90° to 190° F. The scale (in degrees of Fahrenheit) is marked on an ivory back fastened to the tube in

the usual way. It is fitted with a metal collar, fitting the socket, and the part of the tube below the scale should have a length of about 3·5 in. measured from the lower end of the scale to the end of the bulb. The thermometer for ascertaining the temperature of the oil is fitted with collar and ivory scale in a similar manner to the one described. It has a round bulb, a space at the top, and ranges from about 55° F. to 150° F.; it measures from the end of ivory back to the bulb 2·25 in.

METHOD.

(1) *For oils flashing below* 90° *F.*

(1) The test apparatus shall be placed for use in a position where it is not exposed to currents of air or draughts.

(2) The heating vessel or water-bath shall be filled by pouring water into the funnel until it begins to flow out at the spout of the vessel. The temperature of the water at the commencement of the test shall be 130° F., and no heat shall be applied to the water-bath during the test.

(3) The test-lamp shall be prepared for use by fitting it with a piece of flat plaited candle wick, and filling it with colza or rape-oil up to the lower edge of the opening of the spout or wick tube. The lamp shall be trimmed so that when lighted it gives a flame of about 0·15 of an inch diameter, and this size of flame, which is represented by the projecting white bead on the cover of the oil-cup, shall be maintained throughout the test.

When gas is available it may be used conveniently in place of the oil-lamp, and for this purpose a test-flame arrangement for use with gas may be substituted for the lamp.

(4) The bath having been raised to the proper temperature, the oil to be tested shall be introduced into the petroleum cup, being poured in slowly until the level of the liquid just reaches the point of the gauge which is fixed in the cup. In warm weather the temperature of the room in which the samples to be tested have been kept shall be observed in the first instance, and if it exceeds 65° the samples to be tested shall be cooled down (to about 60°) by immersing the bottles containing them in cold water, or by any other convenient method. The lid of the cup, with the slide closed, shall then be put on, and the cup placed into the bath or heating vessel. The thermometer in the lid of the cup has been adjusted so as to have its bulb just immersed in the liquid, and its position shall not under any circumstances be altered. When the cup has been placed in the proper position, the scale of the thermometer shall face the operator.

(5) The test-lamp then shall be placed in position upon the lid of the cup, the leadline or pendulum, which has been fixed in a convenient position in front of the operator, set in motion, and the rise of the thermometer in the petroleum cup watched. When the temperature has reached about 66° the operation of testing shall be commenced, the test-flame being applied once for every rise of one degree, in the following manner :—

The slide shall be drawn open slowly while the pendulum performs three oscillations, and closed during the fourth oscillation.

The pendulum referred to is 24 in. from the point of suspension to the centre of gravity of the weight.

(2) *Oils that have a Flash Point between* 90° *F. and* 120° *F.*

The air-chamber which surrounds the cup shall be filled with cold water to a depth of 1·5 in., and the heating vessel or water-bath filled as usual, but also with cold water. The lamp shall then be placed under the apparatus and kept there during the entire operation, and the temperature of the oil raised at the rate of 2° to 2¼° per minute, the testing being carried out as laid down in the previous section.

CLASS IV.—GAS OIL

(including Special Vaporising Oils, Mineral Colza and Distillate Oils for Heavy Oil Engines).

SPECIFIC GRAVITY.

I.P.T. Serial Designation—G.O. 1.

The apparatus and method shall be as laid down in the special directions for specific gravity (p. 1).

DISTILLATION.

I.P.T. Serial Designation—G.O. 3.

GENERAL.

In general this test is required only for oil for gas making.

APPARATUS.

A distillation flask of 500 c.c. capacity. The flask shall be connected with a condenser and the general arrangements shall be on the same lines to that for the distillation of gasoline (G. 3, p. 5) and kerosene (K. 3, p. 17). The thermometer shall be the high range distillation thermometer (to 400° C.) conforming with K. 3.

METHOD.

Not less than 300 c.c. of the oil shall be distilled at a slow and uniform rate; for 300 c.c. the time for distillation up to 350° C. shall not be less than 45 minutes.

Each 10 per cent. fraction shall be collected separately, and the range over which it is collected stated. The specific gravity of each fraction shall be determined.

Distillation shall be stopped at 350° C., the residue cooled, measured and its specific gravity determined.

TOTAL SULPHUR.

I.P.T. Serial Designation—G.O. 4.
A.S.T.M. Serial Designation D 129—22 T. (modified).

APPARATUS.

The combustion of the oil shall be carried out in a suitable type of bomb calorimeter.

The inner surfaces shall be of materials that are chemically and physically resistant to the process and products of combustion.

The gaskets, insulating materials, etc., shall not be liable to action with the products of combustion in such a way that any increase or decrease of the sulphur content of the liquid in the bomb shall arise.

Particular attention is directed to lead gaskets, which have been found to form lead sulphate if any appreciable area is exposed.

The ignition wire shall be of fine platinum (0·15 mm. diameter is suitable).

Distilled water and all reagents should be sulphur free. If this is found not to be the case a blank shall be made and the results properly corrected.

METHOD.

The weight of oil to be used shall be not less than 1 gram. 10 c.c. of distilled water shall be placed in the bomb. The oxygen pressure shall be not less than 25 atmospheres. The bomb shall be immersed in cold water before firing.

After firing, the bomb shall remain in the water for 10 minutes. This allows practically complete absorption of the oxides of sulphur by the water.

The gases shall be allowed to blow off slowly into the air. The bomb shall be emptied into a beaker and the interior, including cover and all internal parts, washed out thoroughly with distilled water.

If the collected contents and washings are not clear they shall be filtered and the filter paper thoroughly washed with distilled water, the washings being added to the filtrate.

The washings, filtrate, etc., should not exceed 300 c.c.

Precipitation of the Sulphur.—2 c.c. of concentrated hydrochloric acid and 0·5 gram of pure sodium peroxide (Na_2O_2) shall be added, and the solution well boiled.

10 c.c. of barium chloride solution (100 grams $BaCl_2$ $2H_2O$ per litre) shall be boiled in a test-tube and slowly added to the boiling liquors. Boiling shall be continued for at least 15 minutes.

The solution shall be kept on a hot plate or steam-bath for not less than 4 hours. It is recommended that it shall stand all night with suitable cover to prevent undue loss by evaporation.

The paper and precipitate shall be washed with water until free from all traces of chlorides and finally with a solution of ammonium nitrate (10 per cent.) and dried. This treatment assists combustion of the paper and prevents reduction of the barium sulphate. The paper shall be burnt off and the precipitate and paper ash ignited and weighed in the usual manner.

CALORIFIC VALUE.

(*Gross.*)

I.P.T. Serial Designation—G.O. 6.

APPARATUS.

The calorific value shall be determined in a suitable type of bomb calorimeter permitting the use of oxygen at filling-pressures up to 30 atmospheres by the gauge.

The centigrade thermometer employed must be clearly graduated either in 1/100ths of a degree or to every 1/50th of a degree with the graduations sufficiently spaced to allow of estimating the readings to 1/100th of a degree.

A suitable thermometer would have a range of from 12° to 30° C., in 1/50ths, with the lowest graduation 75-80 mm. (3 in.) above the top of the bulb, the total length being about 65 cm. (25 in.). Calorimeter thermometers may be specially tested at the National Physical Laboratory, and certificates, with the corrections as necessary, are issued.

The ignition wire should be of fine platinum, a suitable diameter being 0·15 mm., but if iron wire has to be employed, its weight must be known and allowance must be made for the heat of combustion by 1600 calories per gram.

DETERMINATION OF WATER VALUE OF THE CALORIMETER.

The standard water value of the calorimeter and its parts shall be determined by the combustion of suitable weights of one or more of the following pure compounds and the calorific value for these is to be taken as :—

Naphthalene	..	..	..	..	9620 calories per gram.
Benzoic Acid	..	..	..	..	6330 " " "
Cane Sugar	..	..	..	..	3950 " " "

10 c.c. of distilled water shall be measured into the bomb before the standard water value is determined, and in all estimations a like quantity of distilled water shall be employed in the bomb.

NOTE.—Although not absolutely necessary in determining the calorific value, since the determination of the sulphur in the sample will generally be made from the washings from the bomb after determining the calorific value, the 10 c.c. of water serves to absorb the sulphur trioxide formed, and so it is always convenient to standardise with this quantity of water in the bomb and make all determinations under like conditions.

METHOD.

Not less than 1 gram of oil shall be employed. The requisite quantity of water for the calorimeter shall be weighed out.

The water should be at room-temperature. A sufficient quantity of water for duplicate determinations at least shall be kept in the laboratory near the instrument so that it shall be at the room-temperature when the determinations are to be made.

10 c.c. of distilled water shall be measured into the bomb.

A small strip of filter paper or blotting paper, 4 to 5 mm. wide and of sufficient length when folded into a Λ for both ends to dip into the oil when the strip of paper is laid over the firing wire shall be employed for the ignition.

The weight of this paper shall be accurately determined.

The bomb shall be charged with oxygen to a pressure of not less than 25 atmospheres.

After firing, readings of the thermometer shall be taken every half minute from the time of firing until not less than 12 half-minute readings from the time of reading the maximum temperature have been taken. The rate of loss by radiation during the experiment shall be deduced from the average of the last ten of these readings.

CALCULATION AND EXPRESSION OF CALORIFIC VALUE.

Corrections shall be applied for

(*a*) Losses by radiation.

(*b*) The heat due to ignition-paper.

(*c*) If iron firing-wire is used for its heat of combustion.

No correction is to be made for nitric acid or sulphuric acid formed.

(*a*) *Loss by Radiation.*—To the observed rise of temperature, add :—

(1) For *each* half-minute period during the rise when the mean temperature for the period does not differ by more than 0·5° from the maximum temperature apply the full half-minute radiation correction as determined during the cooling period.

(2) Where the mean temperature for a given half minute is lower by more than 0·5° than the maximum, then for *each* such half-minute period the radiation loss shall be calculated as directly proportional to the mean difference of temperature—*e.g.*, the rate of loss per half minute when the water in the calorimeter is 2·8° above the initial temperature, *i.e.*, is at its maximum, was found to be 0·005°. The mean temperature of the first half minute was 1·4° below the final temperature. The correction for this first half-minute will be :—

$$\frac{1{\cdot}4}{2{\cdot}8} \times 0{\cdot}005 = 0{\cdot}0025.$$

The total radiation correction to be added to the observed rise will be the sum of the individual half-minute corrections up to the time for the maximum reading of the thermometer.

From the weight of water in the calorimeter, *plus* the water equivalent, and from the corrected rise of temperature the total calories shall be calculated.

(*b*) From this value deduct the calories due to the ignition-paper, the calorific value for which is to be taken as 4190 calories per gram.

(*c*) (If iron firing-wire was used, a further deduction for this as 1600 calories per gram shall be made.)

The calories due to the oil alone shall then be calculated and expressed as calories per gram.

Results should also be expressed as British thermal units per pound—B.Th.U.—this value being 1·8 times the calories per gram.

FLASH POINT.

I.P.T. Serial Designation—G.O. 7.
A.S.T.M. Serial Designation—D 93—21 T.

The I.P.T. standard Pensky-Martens closed tester shall be used for determining the flash point of all petroleum products having a flash point above 120° F.

The makers' or vendors' name and an identification number shall be put on each instrument, which shall be certified and marked by the National Physical Laboratory as complying with the dimensions specified.

APPARATUS (FIG. 6).

The Pensky-Martens tester, a diagram of which appears in Fig. 6, shall include the following major parts :—

Cup.—The cup of the I.P.T. standard Pensky-Martens flash tester shall be made of brass and shall satisfy the following dimensional specifications :—

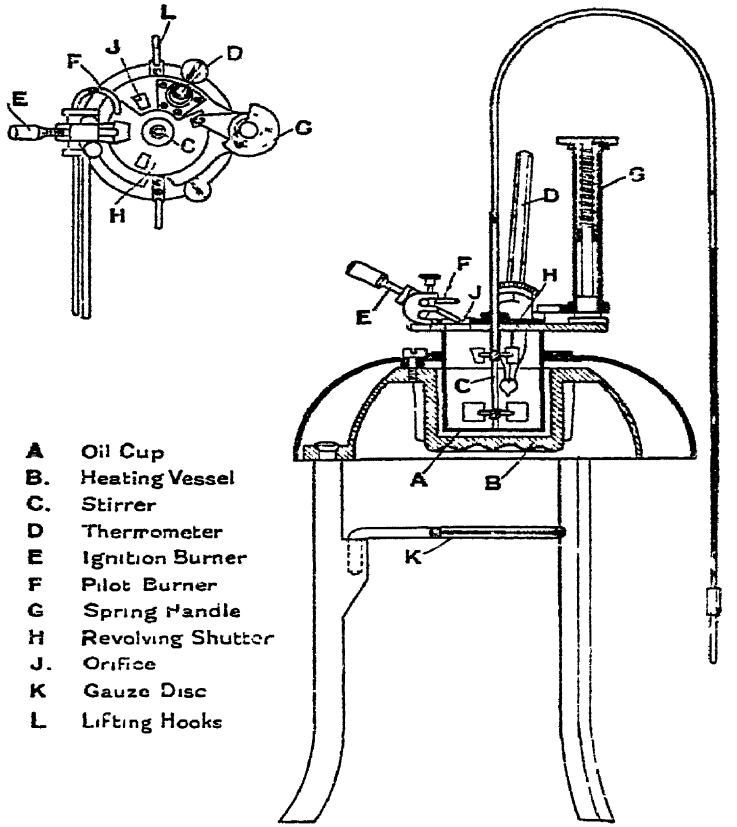


FIG. 6.

THE PENSKY-MARTENS CLOSED TESTER.

Dimensions.	Inches. Normal.	Tolerances.
Inside diameter below filling mark	2·000	±0·050
Difference, inside and outside diameters below filling mark	0·125	±0·005
Inside height	2·200	±0·050
Thickness of bottom	0·095	±0·025
Distance from rim to filling mark	0·860	±0·015
Distance lower surface flange to bottom of cup	·795	±0·015

The inside of the cup may be turned to a slightly larger diameter above the filling mark and the outside may be tapered above the flange, but the wall thickness at the upper edge shall be not less than 0·04 in. (0·1 cm.). The flange should be approximately 0·5 in. (1·27 cm.) wide and approximately 0·125 in. (0·32 cm.) thick. It shall be equipped with devices for locating the position of the lid on the cup and the cup in the stove. A handle, attached permanently to the flange of the cup, is a desirable accessory.

Stirring Device.—The lid shall be equipped with a stirring device consisting of a vertical steel shaft, not less than 0·1 in. (0·25 cm.) nor more than 0·125 in. (0·32 cm.) in diameter, mounted in the centre of the cup, and carrying two two-bladed brass propellers. The blades of both propellers shall be approximately 0·3 in. (0·8 cm.) wide and shall be set at an angle of approximately 45°. The smaller (upper) propeller shall have an over-all diameter of approximately 0·75 in. (1·9 cm.). The larger (lower) propeller shall have an over-all diameter between 1·25 and 1·75 in. (3·175 and 4·445 cm.). The thickness of the propeller blades shall be not less than 0·05 in. (0·146 cm.) nor more than 0·08 in. (0·2 cm.), which limits correspond respectively to No. 17 and No. 14 Birmingham gauge sheet brass. The collars on which the propeller blades are mounted shall have horizontal and vertical dimensions not greater than 0·4 in. (1 cm.).

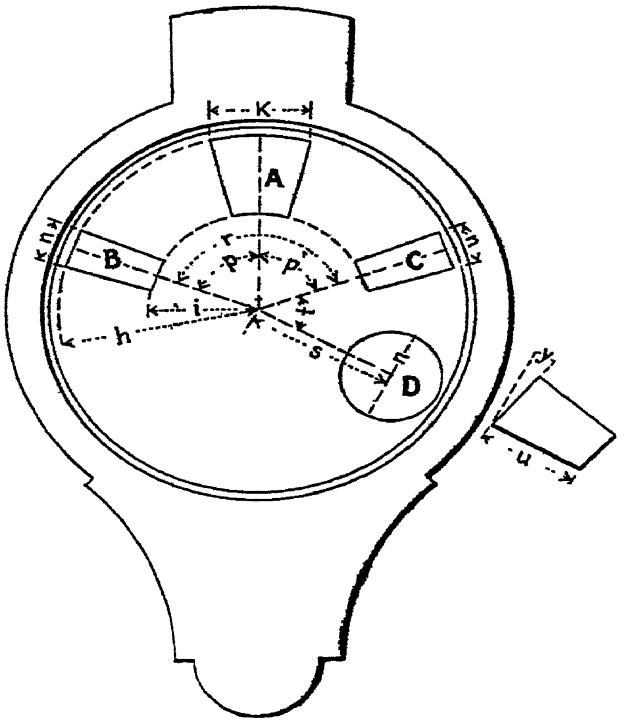
The plane of the centre of the upper propeller shall be 0·4 in. (1 cm.) below the level of the rim of the cup. The plane of the centre of the lower propeller shall be 2·0 in. (5·1 cm.) below the level of the rim of the cup. The level of the rim of the cup is in effect the level of the plane part of the portion of the lower surface of the lid inside the rim.

Cover proper.—The cover proper shall be of brass and shall have a rim projecting downward almost to the flange of the cup and fitting the outside of the cup closely. The thickness of the cover, measured just inside the rim, shall be not less than 0·03 in. (0·08 cm.) nor more than 0·08 in. (0·2 cm.). There shall be a proper locating device engaging with a corresponding locating device on the flange of the cup.

There shall be four openings in the cover, as indicated in Fig. 7.

Opening *A* is an area defined by arcs of two concentric circles and the intersected lengths of two radii. The radius of the outer circle shall be not less than 0·94 in. (2·38 cm.) nor more than 0·97 in. (2·46 cm.). The chord of the arc of the outer circle shall be not less than 0·5 in. (1·27 cm.) nor more than 0·54 in. (1·37 cm.).

Openings *B* and *C* are equal areas, each of the same general form as opening *A* but of approximately half the (angular) width. The radii of the defining inner and outer circles shall be within the limits specified for the radii of the two circles, arcs of which partially define opening *A*. The chord of the outer arc for opening *B* or opening *C* shall be not less than 0·19 in. (0·475 cm.) nor more than 0·22 in. (0·555 cm.). The sum of the areas of openings *B* and *C* shall be not less than 75 per cent. nor more than 100 per cent. of the area of opening *A*. Openings *B* and *C* shall be equally



h	Minimum 0·94 in.,		maximum 0·97 in.		
i	,,	0·53 in.,	,,	0·56 in.	
k	,,	0·50 in.,	,,	0·54 in.	
n	,,	0·19 in.,	,,	0·22 in.	
s	Approximately 0·75 in.				
u	,,	0·5 in.			
Angles *p*	Equal.				
Angle *r*	Minimum 135°,		maximum 140°.		
,, *t*	,,	50°,	,,	60°.	
,, *y*	,,	10°,	,,	15°.	

FIG. 7.

COVER OF PENSKY-MARTENS TESTER.

distant from opening *A* and radii drawn through each of their centres shall be at an angle of not less than 135° nor more than 140°.

Openings *A*, *B*, and *C* need not conform exactly to the shape of geometrical figures bounded by arcs of two concentric circles and intersected lengths of radii. Their boundaries must, however, fall on or between the lines indicated by the limiting values of the dimensional specification of the preceding text and of Fig. 7.

Opening *D* is for a thermometer collar. Its centre is approximately 0·75 in. (1·9 cm.) from the centre of the lid and on a radius at an angle of not less than 50° nor more than 60° from a radius passing through the centre of opening *C*. The thermometer collar shall have an inside diameter of approximately 0·5 in. (1·27 cm.). It shall be set at an angle of not less than 10° nor more than 15° from the perpendicular.

Shutter.—The lid shall be equipped with a brass shutter, approximately 0·1 in. (0·25 cm.) thick operating on the plane of the upper surface of the lid. The shutter shall be so shaped and mounted that it rotates on the axis of the horizontal centre of the lid between two stops so placed that when in one extreme position the openings *A*, *B* and *C* of the lid are completely closed and when in the other extreme position these orifices are completely opened.

(4) *Flame exposure device.*—The flame-exposure device shall have a tip with an opening 0·027 in. (0·7 mm.) to 0·03 in. (0·8 mm.) in diameter. The flame-exposure device shall be equipped with an operating mechanism which, when the shutter is in the "open" position, depresses the tip so that the centre of the orifice is between the planes of the under and upper surfaces of the lid proper at a point on a radius passing through the centre of the larger opening *A* and approximately 0·1 in. (0 254 cm.) from the outer edge of the opening.

NOTE.—A pilot flame for automatic relighting of the exposure flame should be provided.

A bead 0·16 in. (0·4 cm.) in diameter, of some suitable material, may be mounted on the lid so that the size of the test flame can be regulated by comparison.

The mechanism operating the shutter should be of the spring type and constructed so that when at rest the shutter shall exactly close the three openings. When operated to the other extreme the three openings in the lid shall be exactly open and the tip of the exposure tube shall be fully depressed.

Stove.—Heat shall be supplied to the cup by means of a properly designed stove which is equivalent to an air bath. This stove shall consist of (1) an air bath and (2) a top plate on which the flange of the cup rests.

Air bath.—The air bath shall have a cylindrical interior 1·625 in. (4·13 cm.) to 1·66 in. (4·2 cm.) deep and a diameter not less than 0·125 in. (0·32 cm.) nor more than 0·156 in. (0·4 cm.) greater than the outside diameter of the cup. The air bath may be either a flame-heated metal casting or an electric resistance element.

NOTE.—If the heating element is a flame-heated metal casting it shall be so designed and used that the temperature of bottom and walls is approximately the same. On this account it should be not less than 0·25 in. (0·635 cm.) thick. The casting shall be designed so that products of combustion of the flame cannot pass up and in contact with the cup.

If the air bath is of the electric resistance type it shall be constructed so that all parts of the interior surface are heated equally. This necessitates an even distribution of resistance wire over bottom and walls and a method of construction such that heat is given out from the whole core of the resistance element rather than directly from the wire.

Top plate.—The top plate shall be of metal. The total distance from the upper surface of the plate to the bottom of the air bath shall exceed the distance from the under surface of the flange to the bottom of the cup by not less than 0·063 in. (0·160 cm.) nor more than 0·125 in. (0·32 cm.).

The top plate shall be mounted with an air gap between it and the air bath. The top plate may be attached to the air bath by means of three screws and spacing bushings. The spacing bushings should be of proper thickness to define the air gap which shall be not less than 0·125 in. (0·32 cm.) nor more than 0·187 in. (0·475 cm.). The spacing bushings shall be not more than 0·375 in. (0·95 cm.) in diameter.

Thermometers.—Two standard thermometers shall be used with the I.P.T. Standard Pensky-Martens tester. The low range " P. M. " thermometer shall be used for tests when the indicated reading falls within the limits 20 to 200° F. The " P.M. high " thermometer shall be used for tests when the indicated reading falls within the limits 230 to 700° F. For the range 200 to 230° F. either thermometer may be employed, depending on the convenience of the operator. The thermometers shall comply with the specifications given in Table I.

Thermometers shall be mounted securely in the thermometer collar so that from the top of this collar to the bottom of the bulb the distance is 2·2 in. ($\pm$0·05 in.) and so that the bottom of the bulb is 1·5 in. ($\pm$0·1 in.) below the level of the rim of the cup (which corresponds to the level of the lower surface of the portion of the lid inside the rim).

TABLE I.

THERMOMETER SPECIFICATION.

	Low Range "P.M."	High Range "P.M."
Type	Mercury in glass. Solid stem. Nitrogen filled.	Mercury in glass. Solid stem. Nitrogen filled.
Stem	Lead glass or other suitable glass. Enamel back. Diameter, 6 to 7 mm.	Lead glass or other suitable glass. Enamel back. Diameter, 6 to 7 mm.
Bulb	Round. Powell's normal glass, Jena 16 III, or other approved glass. Diameter not to exceed 8 mm.	Round. Powell's normal glass, Jena 16 III, or other approved glass. Diameter not to exceed 8 mm.
Range ..	20° F. to 230° F.	200° F. to 700° F.
Immersion ..	2·2 in. To be marked by a line etched round the stem, or, alternatively, an enlargement made in the stem to ensure 2·2 in. immersion (±0·05″) from the top of the brass plug into which the thermometer is fixed and the bottom of the bulb.	2·2 in. To be marked by a line etched round the stem, or, alternatively, an enlargement made in the stem to ensure 2·2 in. (±0·05″) immersion from the top of the brass plug into which the thermometer is fixed and the bottom of the bulb.
Dimensions ..	Overall length, approximately 275 mm. Distance from bottom of bulb to 20° line, 70 to 85 mm. Distance from bottom of bulb to 230° line, 235 to 250 mm.	Overall length, approximately 275 mm. Distance from bottom of bulb to 200° line, 70 to 85 mm. Distance from bottom of bulb to 700° line, 235 to 250 mm.
Expansion chamber	Required.	Required.
Finish ..	Glass ring.	Glass ring.
Graduation ..	All lines, figures and letters clear cut and distinct. Scale graduated to 1° F. divisions. Every 5° and 10° shall be indicated by longer lines.	All lines, figures and letters clear cut and distinct. Scale graduated in 5° F. divisions. Every 10° to be marked with longer lines than the 5°, and every 50° with longer lines than the 10°.
Figuring ..	The scale shall be figured at each 10° F.	The scale shall be figured at each 50°.
Marking ..	"I.P.T.-P.M.-Low." Identification number. Maker's (or vendor's) name or trade mark.	"I.P.T.-P.M.-High." Identification number. Maker's (or vendor's) name or trade mark.

Each thermometer shall be tested at the National Physical Laboratory, where satisfactory thermometers will be marked with the N.P.L. monogram and year of test, and a certificate of corrections issued.

PROCEDURE.

(3) (*a*)—All parts of the cup and its accessories shall be thoroughly clean and dry before starting the test. Particular care shall be taken to avoid the presence of any gasoline or naphtha used to clean the apparatus after a previous test.

(*b*) The cup shall be filled with the oil to be tested up to the level indicated by the filling mark.

(*c*) The lid shall be placed on the cup and the latter set in the stove. Care should be taken to have the locating devices properly engaged. The thermometer shall be inserted. If it is known that the oil will flash above 220° F. the " P.M. high " thermometer may be selected; otherwise, it is preferable to start with the " Low Range P.M." thermometer and change in case a temperature of 220 to 230° F. is reached.

(*d*) The test flame shall be lighted and adjusted so that it is of the size of a bead 5/32 in. (4 mm.) in diameter.

(*e*) Heat shall be supplied at such a rate that the temperature read on the thermometer increases not less than 9 nor more than 11 degrees per minute. The stirrer shall be turned at a rate of from 1 to 2 revolutions per second.

(*f*) Application of the test flame shall be made at each temperature reading which is a multiple of 2° F. up to 220° F. For the temperature range above 220° F., application shall be made at each temperature reading which is a multiple of 5° F. The first application of the test flame shall be made at a temperature at least 30° F. below the actual flash point. Application of the test flame shall be made by operating the device controlling the shutter and test-flame burner so that the flame is lowered in one half second, left in its lowered position for one second, and quickly raised to its high position. Stirring shall be discontinued during the application of the test flame.

(4) The flash point is taken as the temperature read on the thermometer at the time of the flame application that causes a distinct flash in the interior of the cup. The true flash must not be confused with the bluish halo that sometimes surrounds the test flame for the applications preceding the one that causes the actual flash.

(5) The barometric pressure shall be observed and recorded. No corrections shall be made except in case of dispute when the flash-point figure shall be corrected according to the following rule :—

For each inch (25 mm.) below 29·92 in. (760 mm.) barometric reading add 1°·6 F. to the flash point.

For each inch (25 mm.) above 29·92 in. (760 mm.) barometric reading subtract 1°·6 F. from the flash point.

VISCOSITY.

I.P.T. Serial Designation—G.O. 8.

The Standard Redwood Viscometer No. I. shall be used as laid down under L.O. 8 (p. 45).

CARBON RESIDUE.

I.P.T. Serial Designation—G.O. 9.
A.S.T.M. Serial Designation D 47—21 T.

APPARATUS (FIG. 8).

A. A porcelain or silica crucible, wide form, glazed throughout, 25 to 26 c.c. capacity, 46 mm. diameter.

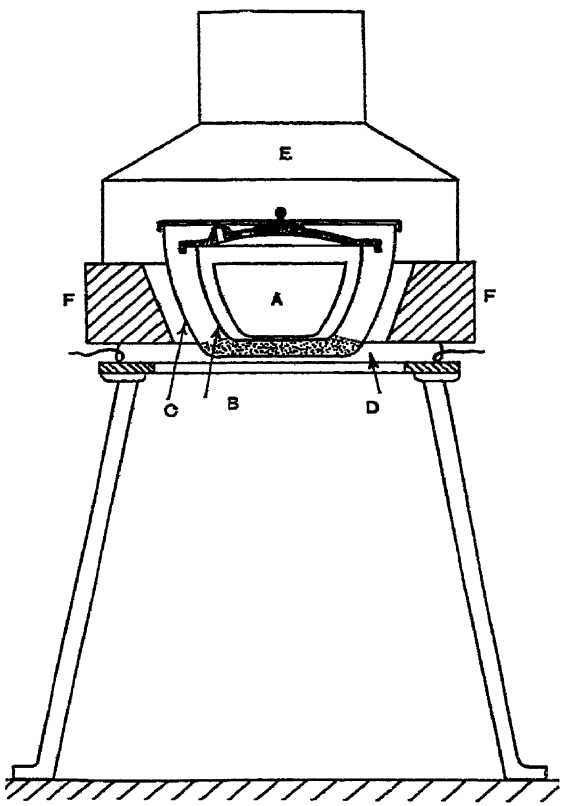


FIG. 8.
CARBON RESIDUE APPARATUS.

B. An iron crucible, 45 c.c. (1½ ounces) capacity, 65 mm. in diameter, 37 to 39 mm. high with cover, the cover being provided with one opening of 5 to 6 mm. diameter.

C. A wrought-iron crucible with cover, about 180 c.c. capacity, 80 mm. diameter, 58 to 60 mm. high. At the bottom of this crucible a layer of sand is placed about 10 mm. deep, or enough to bring the inner iron crucible with its cover on nearly to the top of the wrought-iron crucible.

D. Triangle, pipe stem or silica tube covered, projection on side to allow flame to reach the crucible on all sides.

E. Sheet-iron or asbestos hood provided with a chimney about 2 to 2½ inches high, 2⅛ inches in diameter, to distribute the heat uniformly during the process.

F. Asbestos or hollow sheet-iron block, 6 to 7 inches square, 1¼ to 1½ inches high, provided with opening in centre 3¼ inches in diameter at the bottom and 3½ in. in diameter at the top.

METHOD.

The tests shall be conducted as follows: 10 grams of the oil to be tested shall be weighed in the porcelain crucible A, which is placed in the iron crucible B. These two crucibles are set in the larger iron crucible C, being careful to have the crucible B set in the centre of the iron crucible, covers being applied to each of the iron crucibles. Place on triangle and suitable stand with asbestos block, and cover with sheet-iron or asbestos hood in order to distribute the heat uniformly during the process.

Heat from a Bunsen or other burner shall be applied with a high flame surrounding the large crucible C until vapours from the oil begin to ignite over the crucible, when the heat shall be moderated so that the vapours come off at a uniform rate. The flame from the ignited vapours should not extend over 2 in. above the sheet-iron hood. After the vapour ceases to come off, the heat shall be increased as at the start and kept constant for 5 minutes, making the lower part of large crucible red hot, after which the apparatus shall be allowed to cool somewhat before the crucible is uncovered. The porcelain crucible is removed, cooled in a desiccator, and weighed.

The entire process should require one-half hour to complete when heat is properly regulated. The time will depend somewhat upon the kind of oil tested, as a very thin, rather low flash-point oil will not take as long as a heavy, thick, high flash-point oil.

ASH.

I.P.T. Serial Designation—G.O. 10.

The ash shall be determined by evaporating a suitable quantity of oil to a small volume, transferring to a weighed platinum dish and cautiously continuing the evaporation to dryness, finally igniting until all traces of carbon have disappeared.

The actual quantity of oil to be used is dependent upon the amount of ash, but generally not less than 250 c.c. should be taken.

CLOUD AND POUR POINTS.

I.P.T. Serial Designation—G.O. 11.
A.S.T.M. Serial Designation—D. 97—22 T.

(1)—(*a*) The cloud point of a petroleum oil is that temperature at which paraffin wax or other solid substances begin to crystallise out or separate from solution when the oil is chilled under certain definite specified conditions.

(*b*) The pour point of a petroleum oil is the lowest temperature at which this oil will pour or flow when it is chilled without disturbance under certain definite specified conditions.

(2)—(*a*) The test for cloud point shall be used only for oils which are transparent in layers 1½ in. thick.

(*b*) The test for pour point shall be used for all other petroleum oils and may be used for oils on which the test for cloud point is permitted.

APPARATUS (FIG. 9).

(3) The test jar, A, shall be of clear glass, cylindrical form, flat bottom, approximately 3 cm. in inside diameter and 11·5 to 12·5 cm. high. An ordinary 4-oz. oil sample bottle may be used if the test jar is not available.

(4) The thermometer, B, shall conform to the following specifications.

Type : Etched stem glass.
Total length : Approximately 220 mm.
Stem : Plain front, enamel back, suitable thermometer tubing ; diameter 7 to 8 mm.
Bulb : Powell's Normal. Jena 16 III, or equally suitable thermometric glass ; maximum length, 9·5 mm. ; diameter, not greater than stem.

Actuating liquid : Mercury.
Range : —36° to + 120° F.
Immersion : 11 cm. The words "11 cm. immersion" etched on the stem and also a line around the stem to indicate the depth of immersion.
Distance to −36° line from bottom of bulb : 120 to 130 mm.
Distance to 120° line from bottom of bulb : 195 to 200 mm.
Expansion chamber : To take the mercury at 212° F.
Filled : Nitrogen gas.
Top finish : Plain.
Graduation : All lines, figures and letters clear cut and distinct.
Scale graduated in 2° F. divisions.
Scale numbered every 20° F. starting at −20° F. The −30° line and each succeeding 10° line to be longer than the others.
Special markings : "I.P.T. Cloud and Pour," serial number and the vendor's or maker's name etched on the stem.

Each thermometer shall be tested by the National Physical Laboratory, where satisfactory thermometers will be marked with the N.P.L. monogram and year of test, and a certificate of corrections issued.

(5) The cork, C, shall fit the test jar, and shall be bored centrally to take the test thermometer.

(6) The jacket, D, shall be of glass or metal, shall be water tight, of cylindrical form, flat bottom, about 11·5 cm. deep, with inside diameter approximately 1·25 cm. greater than outside diameter of the test jar.

(7) A disc of cork or felt, 6-7 mm. thick and of the same diameter as the inside of the jacket, is required.

(8) The ring gasket, F, shall be about 5 mm. thick, and made to fit snugly around the outside of the test jar and loosely inside the jacket. This gasket may be made of cork, felt or other suitable material, elastic enough to cling to the test jar and hard enough to hold its shape. The purpose of the ring gasket is to prevent the test jar from touching the jacket.

(9) The cooling bath, G, shall be of a type suitable for obtaining the required temperatures. The size and shape of the bath are optional, but a support, suitable for holding the jacket firmly in a vertical position, is essential. The required bath temperatures may be maintained by refrigeration if available, otherwise by suitable freezing mixtures.

NOTE.—The freezing mixtures commonly used are as follows :—

For temperatures down to				35° F., ice and water.
,,	,,	,,	,,	—5° F., crushed ice and sodium chloride.
,,	,,	,,	,,	—25° F., crushed ice and calcium chloride.
,,	,,	,,	,,	—70° F., solid carbon dioxide and acetone.

The last-named mixture may be made as follows : In a covered metal beaker chill a suitable amount of acetone to 10° F., or lower, by means of an ice-salt mixture. Invert a cylinder of liquid carbon dioxide and draw off carefully into a chamois skin bag the desired amount of carbon dioxide, which through rapid evaporation will quickly become solid. Then add to the chilled acetone enough of the solid carbon dioxide to give the desired temperature.

METHOD.

(10) *Cloud Point.*—The oil to be tested shall be brought to a temperature at least 25° F. above the approximate cloud point. Moisture, if present, shall be removed by any suitable method, as by

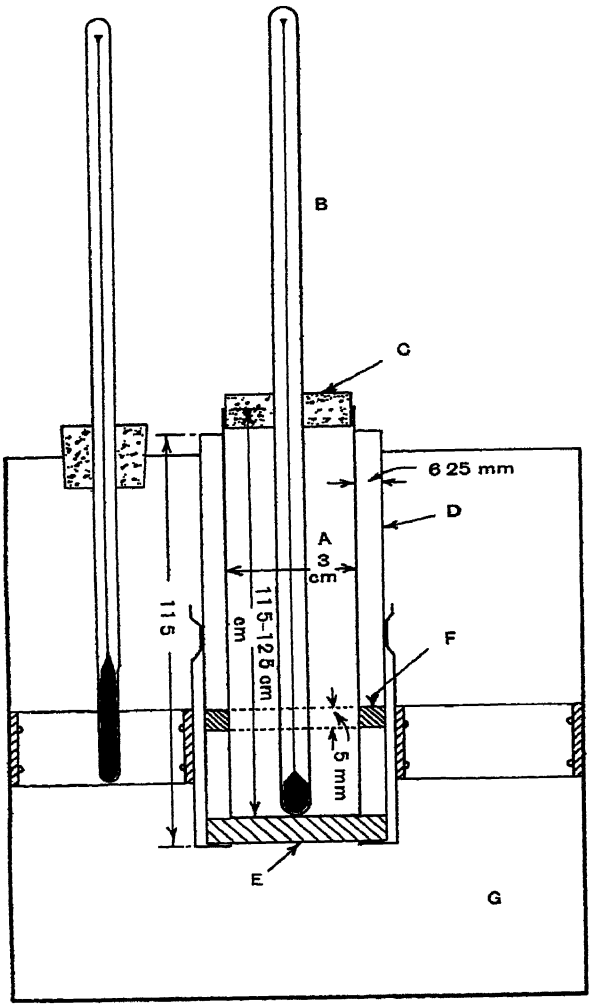


FIG. 9.

CLOUD AND POUR TEST APPARATUS (AS ASSEMBLED FOR CLOUD TEST).

filtration through Plaster of Paris or dry filter paper until the oil is perfectly clear, but such filtration shall be made at a temperature at least 25° F. above the approximate cloud point.

The clear oil shall be poured into the test jar, A, to a height of not less than 2·5 cm., or more than 3 cm. The test jar may be marked to indicate the proper level.

The test jar shall be tightly closed by the cork, C, carrying the test thermometer, B, in a vertical position in the centre of the jar with the thermometer bulb resting on the bottom of the jar.

The disc, E, shall be placed in the bottom of the jacket, D, and the test jar, with the ring gasket, F, 2·5 cm. above the bottom, shall be inserted into the jacket. The disc, jacket and inside of jacket shall be clean and dry.

The temperature of the cooling bath, G, shall be adjusted so that it is below the cloud point of the oil by not less than 15 nor more than 30° F., and this temperature shall be maintained throughout the test. The jacket, containing the test jar, shall be supported firmly in a vertical position in the cooling bath so that not more than 2·5 cm. of the jacket projects out of the cooling medium.

At each test thermometer reading which is a multiple of 2° F., the test jar shall be removed from the jacket, quickly but without disturbing the oil, inspected for cloud, and replaced in the jacket. This complete operation shall require not more than three seconds.

When such inspection first reveals a distinct cloudiness or haze in the oil at the bottom of the test jar, the reading of the test thermometer, corrected for error if necessary, shall be recorded as the cloud point.

(11) *Pour Point.*—The oil to be tested shall be brought to a temperature of 90° F., or to a temperature 15° F. higher than its pour point if this pour point is above 75° F. The oil shall then be poured into the test jar, A, to a height of not less than 5, or more than 5·5 cm. The jar may be marked to indicate the proper level.

The test jar shall be tightly closed by the cork, C, carrying the test thermometer, B, in a vertical position in the centre of the jar with the thermometer bulb immersed so that the beginning of the capillary shall be 3 mm. below the surface of the oil.

The disc, E, shall be placed in the bottom of the jacket, D, and the test jar, with the ring gasket, F, 2·5 cm. above the bottom, shall be inserted into the jacket. The disc, gasket and inside of jacket shall be clean and dry.

After the oil has cooled enough to allow the formation of paraffin wax crystals, great care shall be taken not to disturb the mass of the oil nor to permit the thermometer to shift in the oil. Any disturbance of the spongy network of wax crystals will lead to low and fictitious results.

The temperature of the cooling bath, G, shall be adjusted so that it is below the pour point of the oil by not less than 15 nor more than 30° F., and this temperature shall be maintained throughout the test. The jacket, containing the test jar, shall be supported

firmly in a vertical position in the cooling bath so that not more than 2 cm. of the jacket projects out of the cooling medium.

At each test thermometer reading which is a multiple of 5° F., the test jar shall be removed from the jacket carefully and shall be tilted just sufficiently to ascertain whether the oil around the thermometer remains liquid. As long as the oil around the thermometer flows when the jar is tilted slightly, the test jar shall be replaced in the jacket. The complete operation of removal and replacement shall require not more than 3 seconds. As soon as the oil around the thermometer does not flow when the jar is tilted slightly, the test jar shall be held in a horizontal position for exactly 5 seconds and observed carefully. If the oil around the thermometer shows any movement under these conditions, the test jar shall be immediately replaced in the jacket and the same procedure shall be repeated at the next temperature reading 5° F. lower. In determining the pour point of oils it shall be noted that the first movement of the oil in certain cases is not around the thermometer but from the sides of the test jar. In such cases, this movement shall be considered in making the test. As soon as a temperature is reached at which the oil around the thermometer shows no movement when the test jar is held in a horizontal position for exactly 5 seconds, the test shall be stopped.

The lowest reading of the test thermometer, corrected for error if necessary, at which the oil around the thermometer shows any movement when the test jar is held in a horizontal position for exactly 5 seconds shall be recorded as the pour point. It shall be noted that the pour point is the temperature 5° F. above the solid point or temperature at which the test is discontinued.

CLASS V.—LUBRICATING OIL

SPECIFIC GRAVITY.

I.P.T. Serial Designation—L.O. 1.

The apparatus and method shall be as laid down in the special directions for specific gravity (p. 1).

ACIDITY.

I.P.T. Serial Designation—L.O. 5.

(*a*) *Total Acidity.*—10 grams of the oil shall be weighed into a flask, and 50 c.c. of neutral 95 per cent. alcohol added. The alcohol shall be heated to boiling on a water bath, and the mixture well shaken to ensure solution of the acids in the alcohol.

1 c.c. of 0·5 per cent. solution of phenol phthalein shall be added, and the mixture titrated with $N/10$ potassium hydroxide (free from carbonate).

The results shall be expressed as the number of milligrams of potassium hydroxide necessary to neutralise the acidity of 100 grams of the sample.

(*b*) *Inorganic Acidity.*—100 grams of the oil shall be shaken vigorously in a tap funnel for 1 minute with an equal weight of warm water. The water shall be allowed to separate and then run into a clean flask, cooled and titrated with $N/10$ potassium or sodium hydroxide, using methyl orange as indicator. The result shall be expressed as indicated above.

(*c*) *Organic Acidity.*—This is given by deducting the inorganic acidity from the total acidity.

FLASH POINT (CLOSED).

I.P.T. Serial Designation—L.O. 7.

The Pensky-Martens apparatus shall be employed, as laid down under G.O. 7 (p. 28).

OPEN FLASH POINT AND FIRE POINT.

I.P.T. Serial Designation—L.O. 7a.

APPARATUS.

The oil-cup, heating jacket and thermometer used shall be identical with those employed for the Pensky-Martens closed test (p. 28). The cover of the cup shall, however, be replaced by a clip which encircles the upper rim of the cup and carries the thermometer and test flame.

The thermometers shall be those specified for the Pensky-Marten closed tester.

METHOD.

The quantity of oil used shall be the same as that specified for the closed test, so that closed flash point, open flash point and fire point may be determined in succession with the same sample and with little modification of the apparatus.

The thermometer shall be fixed so that the centre of its bulb is in the vertical axis of the cup and ½ in. below the filling line.

The test flame shall be of the same size as is specified for the closed flash point test and shall be fixed at the vertical axis of the cup and at a level with the upper edge of the cup.

No stirring shall be employed and the greatest precautions must be taken to screen the apparatus from draughts. It is recommended that the test shall be carried out in a fume cupboard free from air currents.

The rate of heating shall be the same as that laid down for the closed test—*i.e.*, 10° F. per minute.

The *flash point* shall be taken as the temperature when a flash appears at any point on the surface of the oil. No correction shall be applied for the exposed mercury thread.

FIRE POINT.

To determine the fire point the heating shall be continued under the above conditions until the oil ignites and continues to burn for 5 seconds. This temperature shall be termed the fire point.

VISCOSITY.

I.P.T. Serial Designation—L.O.8.

GENERAL.

The Redwood No. I. instrument shall be used for all oils the time of flow for 50 c.c. of which, at the temperature of the test, does not exceed 2000 seconds. Oils of greater viscosity are to be tested in a Redwood No. II. instrument.

The Redwood No. I. instrument cannot be satisfactorily employed for measuring viscosities less than twice that of water.

When it is necessary to measure viscosities at temperatures above the boiling point of water, oil shall be used in the heating bath.

It is recommended that viscosity determinations shall be made at one or more of the following temperatures: 70°, 100°, 140°, 200°, 250° F.

REDWOOD NO. I. VISCOMETER.

APPARATUS (FIG. 10).

Oil-cup.—The oil-cup is a cylindrical brass vessel, A, provided with a flange at the base; the upper end is open and at the lower end the bottom of the cup is concave internally to allow of complete drainage of the oil or other liquid with which it may be filled. The flange is provided with a threaded portion, by means of which the oil-cup may be supported in the water-bath. The oil-cup shall be heavily silver-plated internally to reduce the risk of corrosion. The base of the cup is further provided with a tapering central hole, into which the jet is fitted. The level to which the oil is to be filled into the oil-cup shall be indicated by a stout wire, B, fixed into the side of the oil-cup below the oil level; this wire is to be turned upwards at right angles and to be tapered to a sharp point at the precise level for the oil surface.

Dimensions.	Normal. mm.	Tolerance. mm
Internal diameter of oil-cup	46·5 ..	±0·5
Internal depth of cylindrical portion of oil-cup	90·0 ..	±0·25
Height of filling point above upper end of jet (to be adjusted after jet is fixed) .. .	82·5 .. (to top of agate)	±2·00

Jet.—The jet, J, shall be constructed of agate and the central hole of the jet must be drilled and polished internally to the greatest possible precision; it must be truly cylindrical.

The upper end of the jet shall be provided with a concave depression to permit of the introduction of a ball-valve for starting or stopping the flow of oil, while the lower end shall be convex, to prevent the outflowing oil from creeping along the base of the oil-cup.

Dimensions.	Normal. mm.	Tolerance. mm.
Internal length of jet	10·0 ..	±0·02
Internal diameter of jet	1·6 ..	±0·01

The tolerances for the height of the filling point and the internal diameter of the jet are interdependent; a positive tolerance on the height of the pointer must be associated with a negative tolerance on the diameter of the jet or *vice versa*, and be of such magnitude that the time of outflow of an oil from the instrument is in agreement with a standard Redwood viscometer within a limit of ±1 per cent.

Water-bath.—The cylindrical water-bath, approximately 14 cm. diameter, surrounding the oil-cup shall be spun from copper sheet, and shall be provided with a side tube, E, about 3 cm. diameter, by means of which the water or other liquid used may be heated. The side tube shall be brazed into the bath and the junction carefully rounded and cleared from projecting metal to allow of perfectly free circulation of the heated liquid. All joints in the side tube shall be brazed. The copper bath shall have a stout brass ring brazed into its base. The ring shall be threaded internally to take the screwed flange of the oil-cup. The joint between the oil-cup and the brass ring shall be made by a leather or asbestos washer. The upper rim of the copper bath shall be turned over to avoid a sharp edge. A tap, D, shall also be provided to allow of the bath being emptied.

As an alternative method of heating, the side tube may be dispensed with, and the bath wound externally with suitable resistance-wire for electrical heating. Efficient lagging on the exterior shall be provided.

Stirrer.—The stirring of the bath shall be effected by means of a cylinder surrounding the oil-cup, provided with 4 vanes, H, the upper and lower portions are to be turned in opposite directions. A curved shield shall be fixed to the upper edge of the cylinder and shall extend to within approximately 1·2 cm. of the walls of the bath. This provides a means of attachment for the handles used for rotating the stirrer, and a support for the bath thermometer. The cylinder supporting the vanes shall be cut away between the points of attachment of the vanes to allow of more ready transference of the heat from the liquid in the bath to the oil-cup. Vertical grooves shall also be provided on the inner surface of the stirrer cylinder to prevent the liquid in the bath being carried into the oil-cup when the stirrer is lifted.

Valve.—The valve, V, for the starting and stopping of the flow of the liquid from the oil-cup shall consist of a metal ball, approximately 11 mm. diameter, carried on a stiff wire, No. 16 S.W.G. (1·63 mm. diameter); both wire and ball shall be heavily silver-plated. The upper end of the wire shall be bent to provide a hook by means of which the valve may be hung on the side of the oil-cup while the oil is flowing through the jet.

Thermometer Support.—A spring clip, S, to support the oil-cup thermometer, T, shall be provided. The width of this clip shall be 20 mm. ($\pm$0·5 mm.). This is carried on a vertical rod, R, riveted to the upper edge of the oil-cup. This support shall be so arranged as to ensure that the bottom of the thermometer bulb shall be 65 to 70 mm. below the rim of the oil-cup.

Oil-cup Cover.—A brass cover in two parts shall be provided to fit the upper end of the oil-cup. Each half must be fitted with insulating handles for convenience in lifting. Suitable holes are to be provided for the oil-cup thermometer and valve-wire to pass through.

Stand.—An iron tripod-stand of suitable height with levelling screws shall be provided to support the bath.

Screen.—A suitable screen shall be provided around the stand to prevent undue cooling of the under side of the instrument when it is used for tests above 100° F.

Level.—A circular level, mounted on a brass plate, to fit the upper end of the oil-cup shall be provided.

THERMOMETER SPECIFICATION.

Type: Mercury in glass; solid stem; nitrogen filled.

Stem: Lead glass, or other suitable glass. Enamel back Diameter, 5·5 to 6·5 mm. A swelling shall be provided approximately 90 mm. from the bottom of the bulb and immediately below the first graduation, to prevent slipping in the clip.

Bulb: Cylindrical. Powell's normal. Jena 16 III, or other approved glass. Approximate length, 18 to 20 mm.

Range: (*a*) Low range, 30° to 150° F.
(*b*) High range, 130° to 250° F.

Immersion: 65 mm.

Dimensions: The distance from the bottom of the bulb to the lowest graduation mark shall be approximately 105 mm.

Expansion chamber: Required.

Finish: Glass ring.

Graduation: Every $\frac{1}{2}$° F. Each degree shall not be less than 1·5 mm. Each 5° and each 10° shall be indicated by longer lines.

Figuring: The scale shall be figured at each 10°.

Marking: "I.P.T. Redwood Viscometer." Identification number. Maker's (or vendor's) name or trade mark.

Each thermometer shall be tested at the National Physical Laboratory, where satisfactory thermometers will be marked with the N.P.L. monogram and year of test, and a certificate of corrections issued.

(*b*) *Bath thermometers.*—Shall be similar in construction and range to oil-cup thermometers except that dimensions of stem and bulb need not be adhered to so closely, and shall be graduated at every 1° F. The bath thermometer shall be fitted into a brass collar to enable it to be supported by the stirrer shield. The collar shall be fixed just below the lowest division of the scale.

Each standard Redwood viscometer No. I. shall be marked with the maker's (or vendor's) name and a serial identification

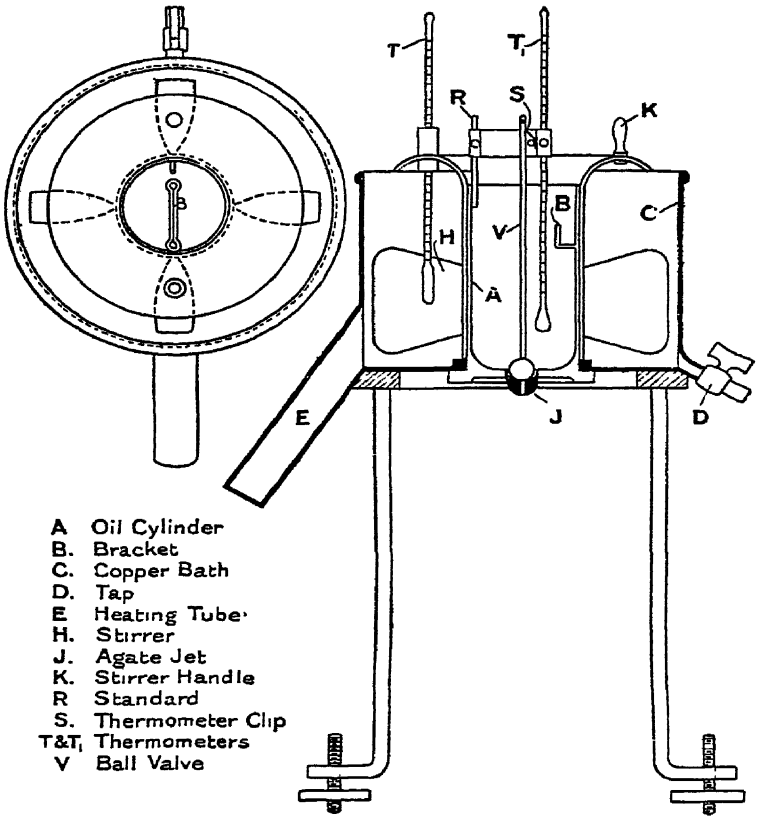


FIG. 10.

REDWOOD NO. I. VISCOMETER.

number. Each instrument shall be submitted to the National Physical Laboratory and its compliance with the dimensions laid down in this specification checked. It will be subjected also to a flow test in comparison with a standard reference viscometer at the Laboratory and must agree within ±1 per cent. of the time

of flow of the reference viscometer. Instruments complying with this specification and being of the required accuracy will be certified by the N.P.L.

55 *cc. Flask.*—This shall conform to the National Physical Laboratory Class A, and shall be marked accordingly.

METHOD.

To carry out a viscosity determination the oil-cup of the viscometer shall first be cleaned with a suitable solvent (ether, benzene, petroleum spirit, etc.) and shall be thoroughly dried to remove all trace of the solvent employed. If it is necessary to use any material for rubbing the interior of the oil-cup or for cleaning the jet, soft tissue paper should be employed. No material likely to leave fluff in the oil-cup shall be used. The apparatus shall be set up and levelled. To eliminate hysterisis, in standard determinations the oil shall be heated for 1 hour at 212° F., allowed to cool and be maintained at 60° F. (±10° F.) for not less than 24 hours before testing.

The oil, whose viscosity is to be determined, shall be heated in a separate vessel to within a degree or two of the temperature at which the test is to be made. The bath of the viscometer shall be heated up to a few degrees above the required test-temperature and the oil poured into the oil-cup through a filter of very fine-meshed gauze. The temperature of the bath shall be adjusted until the oil in the cup is maintained at the desired value, the stirrer in the bath being gently rotated at frequent intervals, or preferably continuously, and the oil gently stirred during the preliminary period. The oil shall not be stirred during the actual test. When the temperature of the oil has become quite steady at the desired value, the adjustment of the oil level shall be made by allowing the oil to flow out until the point of the level adjusting mark is seen to touch the under surface of the oil. The oil-cup cover shall be put in position and the test commenced. A clean, dry 50 c.c. flask is placed centrally below the jet, the neck of the flask being adjusted to within a few millimetres of the underside of the jet. For tests at temperatures above 70° F. the flask shall be supported in a beaker packed with cotton wool or other heat-insulating material to prevent undue cooling during the run. The valve is then lifted and a stopwatch simultaneously started. The watch shall be stopped at the instant the oil reaches the graduation mark of the flask and a final reading of the oil-cup thermometer noted. The test shall be considered invalid if the temperature of the oil varies during the test by more than half a degree for temperatures below 150° F., or by more than 2 degrees for temperatures above 150° F.

EXPRESSION OF RESULTS.

Results are to be expressed as: (1) Time in seconds at the temperature of the test. (2) In absolute units (centimetre-gramme-second units), the following formula being employed.

$$\frac{V}{D}=AT-\frac{B}{T}$$

V = Viscosity of the oil.
D = Density at temperature of test.
T = Time (seconds) for flow of 50 c.c.
A and B are instrument constants. The value for A with the standard viscometer is 0·0026; that of B is 1·7.

REDWOOD No. II. VISCOMETER.

The Redwood No. II. instrument shall be used for all oils, the time of flow for 50 cc. of which in the Redwood No. I. Viscometer exceeds 2000 seconds.

The agate tube of the Redwood No. II. Viscometer is of such dimensions that the time of efflux of 50 c.c. of oil in the instrument is approximately one-tenth the time in a No. I. Viscometer.

APPARATUS (FIGS. 11 AND 12).

REDWOOD NO. II. VISCOMETER.

Oil-cup.—The dimensions of the oil-cup proper (A) shall be the same as those for the Redwood No. I. Viscometer. The base of the cup is prolonged as a brass tube (B), which carries the agate jet (C). The level to which the oil is to be filled into the oil-cup shall be indicated by a stout wire, riveted into the side of the oil-cup below the oil level; this wire shall be turned upwards at right-angles and tapered to a sharp point at the precise level of the oil. The oil-cup shall be heavily silver-plated internally to avoid corrosion.

Dimensions	Normal. mm.	Tolerance. mm.
Internal diameter of oil-cup	46·5 ..	±0·5
Internal depth of cylindrical portion of oil-cup	90·0 ..	±0·5
Height of filling point above upper end of jet (To be adjusted after jet is fixed)	82·5 .. (to top of agate)	±2·5

There is a tube extension at the bottom of the oil-cup to hold the jet. This tube shall be approximately 25 mm. outside diameter, and bore suitable to the agate jet, approximately 10 mm. A screw connection is provided into a lower metal tube (D), which is suitably

attached to the bottom of the heater bath of the instrument. A recess is provided by this tube below the agate jet, approximately 20 mm. deep and 17·5 mm. diameter.

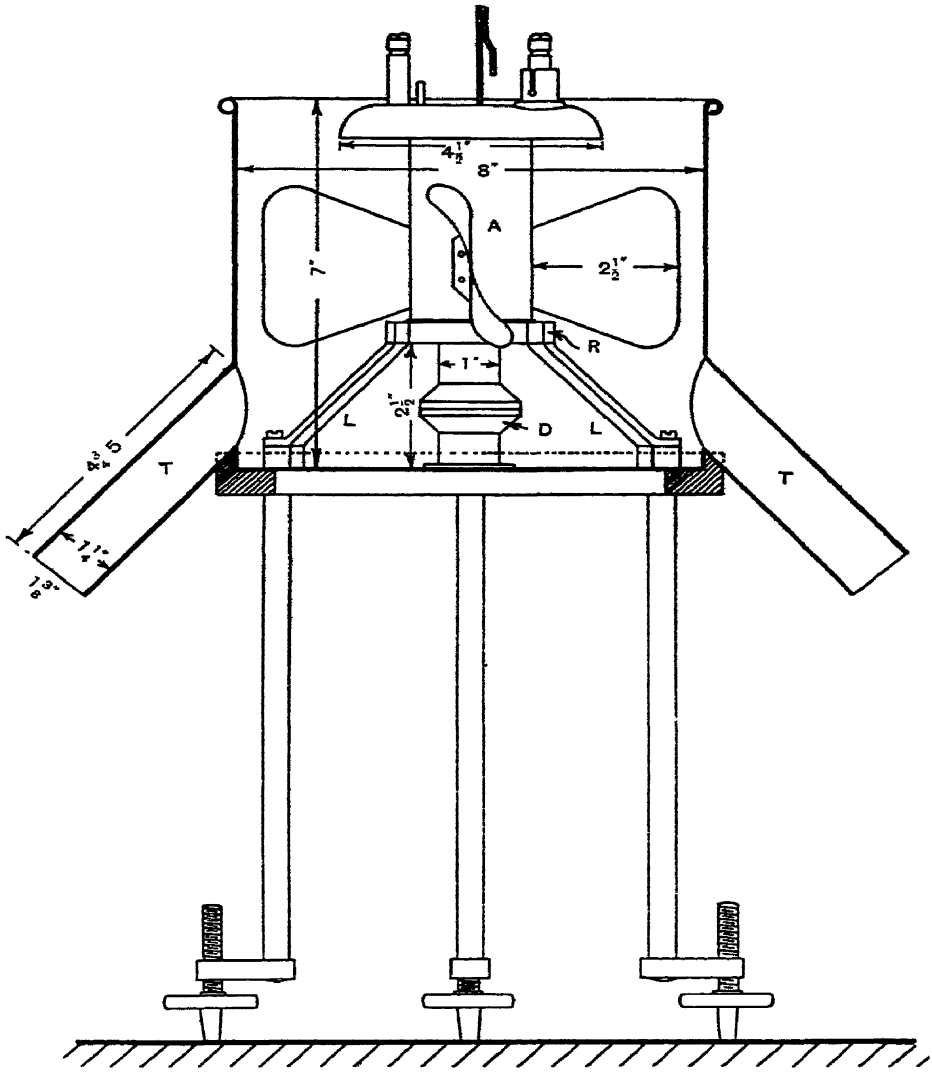


FIG. 11.

The oil-cup shall be held securely in position by a brass ring (R) around the bottom of the cup, this ring being carried on three legs (L) set at an angle and fastened by screws to blocks in the bottom of the heating vessel.

The distance from the underside of the oil-cup proper to the bottom of the heating bath shall be approximately 35 mm.

Jet.—The jet shall be fixed firmly in position in the metal tube extension of the bottom of the oil-cup. The jet shall be constructed of agate and the central hole of the jet must be drilled and polished

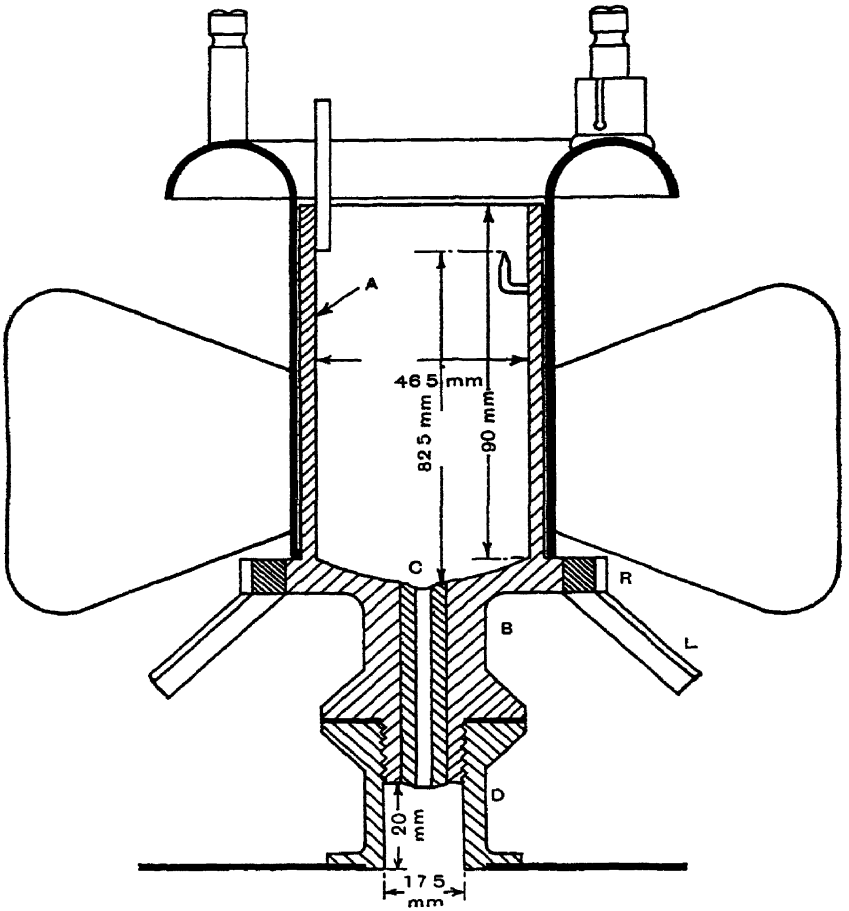


FIG. 12.

REDWOOD NO. II. VISCOMETER—SECTION OF CUP.

internally to the greatest possible precision; it must be truly cylindrical. The upper end of the jet shall be provided with a concave depression to permit of the introduction of a ball-valve for starting or stopping the flow of oil, while the lower end shall

be convex, to prevent the outflowing oil from creeping along the base of the oil cup.

Dimensions.	Normal.	Tolerance.
	mm.	mm.
Internal length of jet	50·0 ..	±0·02
External diameter of jet (approximately) ..	10·0	

The diameter of the bore of the jet shall be such that by adjustment of the height of the filling point within the permitted tolerance the time of flow for 50 cc. of oil is one-tenth (± 4 per cent.) that of the same oil at the same temperature in a Redwood No. I. Viscometer, the oil used for standardising having a time of approximately 4000 seconds for 50 cc. in Redwood I.

Water-bath.—The bath for the heating liquid shall be of copper, 0·05 to 0·06 in. thick (16 or 17 B.G.) and shall measure approximately 20·5 cm. in diameter, and 18 cm. in depth. It shall be provided with a tap for emptying and one (or two) side tubes (T), approximately 3·2 cm. diameter, for heating the contents. If two side tubes are provided they shall be on opposite sides of the bath.

Stirrer.—This shall be identical in construction with the stirrer for the Redwood No. I. Viscometer. The curved shield at the top, fitted to avoid splashing of the bath contents into the oil, shall be the same diameter as in the No. I. Viscometer, *viz.*, approximately 11·5 cm. outside diameter. The vanes on the stirrer shall, however, be larger, and have an approximate width of 6·5 cm.

Valve, Oil-cup cover, thermometer support, stand, screen and level, shall be as laid down for the Redwood No. I. Viscometer.

Thermometers.—(*a*) *Oil cup.*—These shall conform to the specification for the "low" range and "high" range oil-cup thermometers for the Redwood No. I. Viscometer.

(*b*) *Bath.*—This shall conform to the specification for the bath thermometer for the Redwood No. I. Viscometer.

Each Standard Redwood Viscometer No. II. shall be marked with the maker's (or vendor's) name and a serial identification number. Each instrument shall be submitted to the National Physical Laboratory and its compliance with the dimensions laid down in this specification checked. It will also be subjected to a flow test in comparison with a Standard Reference Viscometer at the Laboratory and must agree within ± 4 per cent. of the time of flow of the Reference Viscometer.

Instruments complying with this specification and being of the required accuracy will be certified by the N.P.L.

METHOD.

Determination of viscosity shall be carried out in accordance with the procedure laid down on p. 49 for the Redwood No. I. Viscometer.

CARBON RESIDUE.

I.P.T. Serial Designation—L.O. 9.
A.S.T.M. Serial Designation—D 47—21 T.

The apparatus and method shall be as laid down under G.O. 9 (p. 36).

COLD AND POUR POINTS.

I.P.T. Serial Designation—L.O. 11.
A.S.T.M. Serial Designation—D 47—22 T.

The apparatus and method shall be as laid down under G.O. 11 (p. 38).

HARD ASPHALT.

I.P.T. Serial Designation—L.O. 12.

The method shall be as laid down under F.O. 12 (p. 69).

SOFT ASPHALT.

I.P.T. Serial Designation—L.O. 13.

The method shall be as laid down under C.P. 13 (p. 98).

WATER.

I.P.T. Serial Designation—L.O. 14.
A.S.T.M. Serial Designation—D. 95—23T.

The apparatus and method shall be as laid down under F.O.14 (p. 70).

SAPONIFIABLE MATTER.

I.P.T. Serial Designation—L.O. 15.

Two methods, one volumetric and one gravimetric, are specified. The volumetric method is recommended for rapid determination of approximate accuracy when the nature and approximate saponification value of the saponifiable oil are known. The gravimetric method is more tedious but is applicable and shall be used when the nature of the fatty oil is unknown.

Volumetric Method.—A sufficient quantity of the sample to represent not more than 2 grams of fatty matter, and not more than 10 grams in all, shall be weighed into a 250 c.c. flask and 25 c.c. of approximately seminormal alcoholic potash (prepared from rectified spirit of specific gravity not over 0·810) added. At the same time 25 c.c. of alcoholic potash shall be introduced into a similar flask of the same type of glass, with the usual precautions to ensure that the volume of alcoholic potash is the same in each case to the nearest drop.

The solutions shall be boiled under a reflux condenser for 1 hour, with frequent agitation, and, after cooling, each shall be titrated with seminormal hydrochloric acid, using phenolphthalein as indicator.

From the difference in quantity of acid required for the blank and assay tests, together with the known or assumed saponification value of the fatty matter, the percentage of saponifiable matter is calculated.

The following saponification values shall be used as the basis of calculation :—

Arachis Oil	190	Tallow	195
Castor Oil	180	Sperm Oil	130
Olive Oil	190	Wool Wax	100
Rape Oil	175	Blown Cotton Seed Oil ..	220
Lard Oil	195	Blown Rape Oil	200

Gravimetric Method.—The product after saponification has been carried out as described above shall be transferred to a wet tap funnel, using about 30 c.c. of water and 30 c.c. of petroleum ether (boiling point below 80° C.) to rinse the flask. The mixture is shaken, allowed to separate and the lower layer drawn into a second tap funnel.

The separated soap solution shall be shaken with further charges of petroleum ether until free from oil, and finally evaporated to dryness in a porcelain dish heated on a steam bath, redissolved in water, boiled, acidified with a slight excess of 20 per cent. sulphuric acid and heated until the fatty acids separate.

The petroleum ether solution of the mineral oil in the first

funnel shall be washed until free from soaps by shaking with successive small charges of water, and the washings in turn shaken with the petroleum ether in the second funnel and added to the main soap solution prior to evaporation to dryness. During the washing operations a small proportion of alcohol may be added to break up obstinate emulsions, but care must be taken that the concentration of alcohol does not at any time rise high enough to take mineral oil into solution. This is particularly liable to occur when large amounts of soaps are present and the concentration of alcohol should not be allowed to exceed 20 per cent.

The petroleum ether solutions shall be transferred to a weighed flask, care being taken to reject any drops of water, the petroleum ether distilled off on a steam bath and the flask and residual oil heated in the steam-oven until of constant weight. This gives the percentage of non-volatile unsaponifiable oil (including waxes and certain alcohols).

The fatty acids separated from the soap solution shall be extracted with methylated ether and washed free from mineral acid. This solution is distilled and heated till of constant weight as described above. This gives the percentage of non-volatile fatty acids in the sample. When the fatty acids are known to have been present originally as neutral glycerides it is sufficient to multiply the percentage found by 1·05 to obtain the percentage of saponifiable matter present.

In cases where a precipitate of asphaltic matter is noticed in either the ether or the soap solution it shall be filtered out, washed, weighed and added to the weight of unsaponifiable oil.

When the unsaponifiable oil preponderates in quantity or volatilises appreciably at 100° C., the direct estimation of fatty acids is to be used in determining the proportion of saponifiable matter. When the saponifiable oil preponderates the direct estimation of unsaponifiable oil is to be used, but in either case due precautions must be taken to avoid misleading results due to loss by evaporation. Normally, such loss, as ascertained by totalling the directly-determined percentages of unsaponifiable oil and fatty matter, will not exceed 1·5 per cent. including manipulation losses.

When a very small percentage (*e.g.*, 0.2 per cent.) of saponifiable matter is found, it should be remembered that this may be due to natural petroleum acids, and not added fatty matter.

The test as above described does not give accurate results when animal or vegetable waxes are present. The higher alcohols which are present in large proportions will, in the course of the estimation, be included with the unsaponifiable matter. The analysis of oils containing such waxes is, therefore, a matter too complex for reduction to the terms of a standard test.

DEMULSIFICATION NUMBER.

I.P.T. Serial Designation—L.O. 16.

A.S.T.M.—R.E. Test (1922). D. 157—23 T.

1. The Demulsification Number is the number of minutes required for an oil to separate when emulsified and separated under definitely prescribed conditions.

APPARATUS (FIG 13).

2. *Steam Generator.*—The steam generator (G) shall be made of either metal or glass of at least one litre capacity, capable of withstanding heat necessary for continued use in production of steam. It shall be fitted with three outlets with suitable connections for rubber tubing. In case of a metal generator, a large opening for filling and suitable water gauge shall be provided.

3. *Baths.*—Baths shall be of either metal or glass with a capacity of 3 to 3·5 litres and a depth of 8½ to 9 in. A good quality battery jar or beaker is entirely satisfactory. A glass bath shall be used for emulsification, and a metal or glass bath may be used for the separation.

4. *Source of Heat.*—Heat for the steam generator shall be supplied by a suitable gas burner or electric hot plate. The separating bath (H) may be heated by any convenient means, including an auxiliary steam line (N) as shown in Fig. 13.

5. *Oil Container.*—The oil container (D) shall be a 25 by 200-mm. test tube or special cylinder graduated from 0 to 50 c.c. in cubic centimetres, each even 5 c.c. graduation line to encircle the tube.

6. *Steam Pipe.*—Steam pipe or steam delivery tube shall consist of a piece of glass tubing exactly 2·5 mm. in inside diameter and 30 cm. long. The steam pipe shall be cut off diagonally at an angle of 30 degrees with the axis of the tube at the discharge orifice, and shall be bent at a right angle 25–26 cm. from the discharge orifice.

7. *Accessories.*—Accessories shall consist of :—

(*a*) Suitable wooden or metal frames or holders for holding oil containers in a vertical position in the baths.

(*b*) Thermometers for separating and emulsifying baths (floating type thermometers of suitable range).

OIL THERMOMETER SPECIFICATION.

Type.—Mercury in glass. Solid stem.

Range.—30° to 212° F.

Immersion.—Total.

Dimensions.—The distance from the bottom of the bulb to the 30° mark shall be approximately 35 mm., and to the 190° mark 210 m.m.

Finish.—Glass ring.

Graduation.—Every degree. Every 5° and 10° shall be indicated by longer lines.

Figuring.—The scale shall be figured at each 10°.

Marking.—"I.P.T. Demulsification." Identification number. Maker's (or vendor's) name or trade mark.

Thermometers shall be tested at the National Physical Laboratory under Class B regulations.

(*d*) Corks, rubber tubing and screw pinch cocks.

METHOD.

8. *Preparation.*—The apparatus shall be assembled as shown in Fig. 13. The steam generator shall be filled half full of water and heat applied. Baths shall be filled with 3 litres, (±60 c.c.) of water. The temperature in the separating bath (H) shall be raised to and maintained at 200 to 203° F.

The temperature of the emulsifying bath (E) shall be brought to 67° F. at the start of the test, and is not controlled thereafter. Twenty cubic centimetres of the oil to be tested shall be measured in the oil container at room temperature and the latter placed in the holder of the emulsifying bath. The steam pipe, or delivery tube, shall be connected to the steam generator with suitable rubber tubing, and screw pinch-cocks placed as shown in Fig. 13.

Care shall be taken to see that the apparatus, particularly the oil container, oil container thermometer and the steam delivery tube, are *chemically clean* before using. Care shall also be taken to prevent any foreign materials from entering the steam generator as any contamination of the steam renders the test valueless.

9. *Emulsification.*—The steam delivery tube line shall be steamed out until condensation disappears. A cork having two openings. with the thermometer in one, shall be placed in the mouth of the oil container. The thermometer shall be adjusted so that the bottom of the bulb is 2–2·5 cm. from the bottom of the oil container. The steam delivery tube shall be inserted through the second opening in cork. (Note—this fitting shall be loose) so that the end of the steam delivery tube shall touch the centre of the bottom of the oil container. Steam shall be admitted at a rate that will maintain the temperature of the oil, as shown on the thermometer in the oil container, between 190 and 195° F. (NOTE.—The usual time necessary for the temperature of the oil to come to this point

is 45 to 75 seconds, depending on its character.) This control shall be effected by manipulation of the pinch-cocks on the steam delivery line and steam exhaust line from the steam generator. Steam supply shall be sufficient at all times to cause a generous discharge from the exhaust line. Steaming shall be continued until the volume of condensed steam and oil in the oil container tube is 40 c.c.

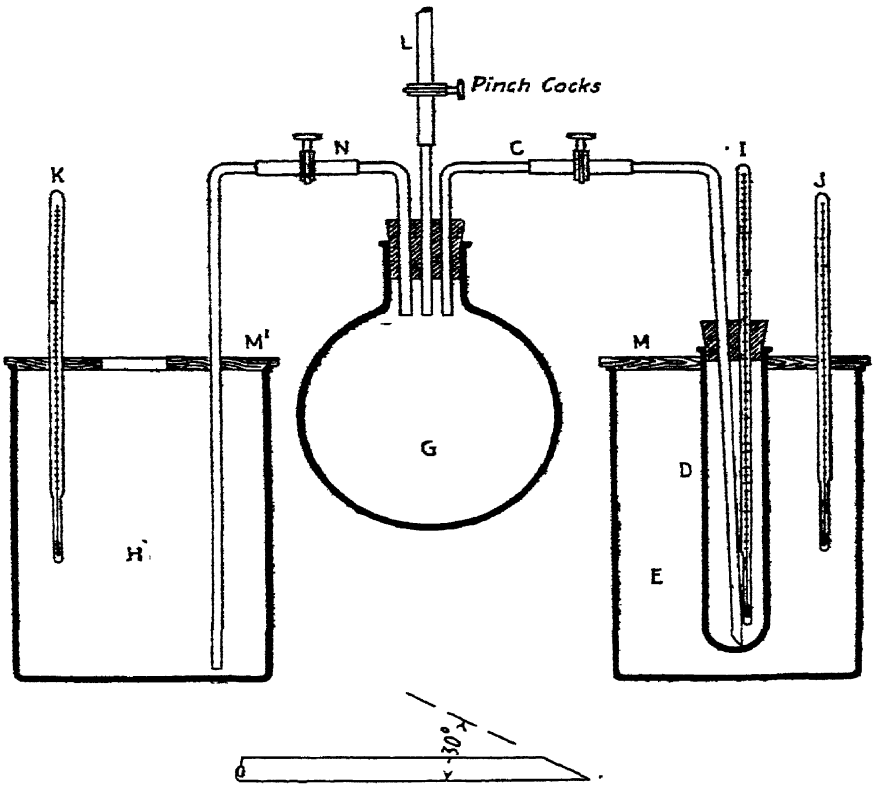


FIG. 13.

APPARATUS FOR TEST FOR RESISTANCE TO EMULSION.

±3 c.c. The time required for this operation shall be 4 to 6·5 minutes, depending on the quality of the oil, altitude, etc. If condensed water amounts to 20 c.c. in less than 4 minutes, it shall be taken as an indication of wet steam or incomplete steaming out of the line, and the test shall be re-run.

NOTE.—The apparent volume in the tube near the end of the steaming operation is approximately 12 to 15 c.c. greater than the actual volume due to displacement caused by steam, thermometer and steam delivery tube.

10. *Separation.*—The steam delivery tube shall be withdrawn as soon as the required volume is obtained. The oil container and holder shall be transferred immediately to the separating bath which shall be maintained at 200 to 203° F. It is extremely important that the temperature of the separating bath be maintained within the given limits. A stop watch shall be started at the instant of withdrawal of the steam delivery tube. The cork containing the thermometer shall be removed after the oil container and contents have been placed in the separating bath. The contents of the oil container shall be examined every 30 seconds, and the volume of the separated oil layer shall be recorded. This examination shall be made by withdrawing the oil container and its contents from the separating bath, and reading the volumes.

This operation shall not require over five seconds. No differentiation shall be made between clear and turbid oil.

This reading operation shall be continued until 20 c.c. of oil has separated. In cases where the 20 c.c. oil layer does not separate in 20 minutes, the test shall be discontinued at this point.

In cases where the interface between more-or-less clear oil and the emulsion is not a clear, straight horizontal line, the position of such a line is carefully estimated to the nearest 0·5 c.c. If, at the end of 20 minutes, there is not a well-defined horizontal interface, then, and then only, the contents of the oil container may be stirred for two seconds with a clean glass rod to obtain a better line of demarcation.

11. *Interpretation.*—In interpreting results, the assumption shall be made that the rate of emulsion separation is directly proportional to the resistance to emulsification of the tested oil. In order to establish a basis for comparison, a definite value of one shall be assigned to an oil which, under conditions of the test, separates completely from emulsion in one minute. This is equivalent to an emulsion separation rate of 0·33 c.c. per second or 20 c.c. per minute.

NOTE.—The expansion of the oil at the higher temperature is disregarded, so when the separation of the oil is complete, as regards the interface between the oil and emulsion layers, the volume of separated oil will always be more than 20 c.c. This in no way interferes with the reproducibility of results; and the time shall be recorded when 20 c.c.—and not necessarily all—of the oil have separated, when this occurs in less than 20 minutes.

12. *I.P.T. Demulsification Number.*—Results shall be reported, in minutes and half minutes, of the time necessary for complete (20 c.c.) oil separation. The time in minutes and half minutes shall be reported as the "Demulsification Number, I.P.T. Method." In cases where the required volume shall not have separated in 20 minutes, the oil shall be reported as having a Demulsification number of 20 plus. If duplicate determinations vary by more than

one minute, a third test shall be made and the average of the three tests reported.

NOTE.—Oils which emulsify will separate into three layers: *top*, clear or turbid oil; *middle*, lacey or creamy emulsion; *bottom*, clear or milky water. The I.P.T. Demulsification Number is derived from the top layer.

With care and proper attention to details, duplicate determinations should not differ by more than 0·5.

VOLATILITY.

I.P.T. Serial Designation—L.O. 17.

B.E.S.A. Specification No. 148—1922.

APPARATUS.

The apparatus used is illustrated in Fig. 14. It consists of a vapour bath constructed of 1·32 in. (approximately 0·8 mm.) sheet copper. The trough running longitudinally down the bath is 13 in. (330 mm.) long by $1\frac{5}{8}$ in. (41 mm.) deep by $2\frac{3}{8}$ in. (60 mm.) wide, and the " D " shaped portion forms a jacket surrounding the trough. Two holes, 1 in. (25 mm.) and $\frac{3}{4}$ in. (19 mm.) diameter, respectively, on opposite sides of the trough and at opposite ends of the vapour jacket, are fitted with tubes projecting to a height

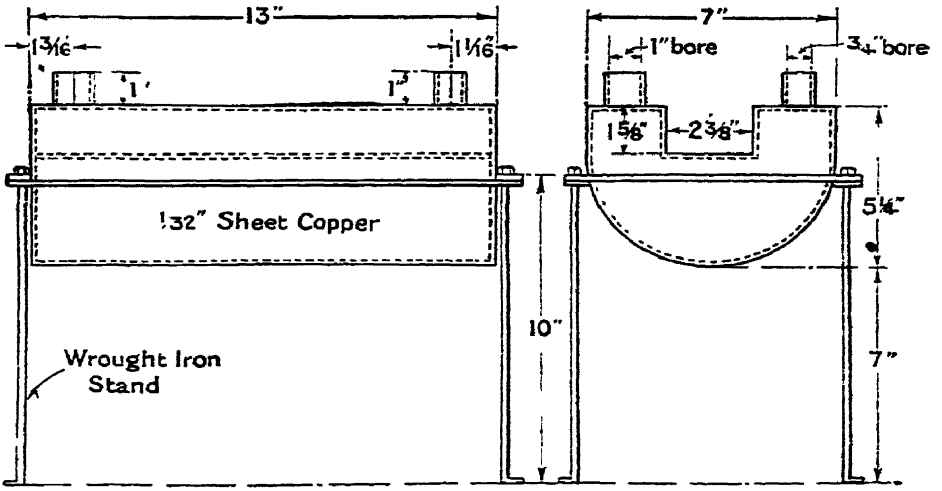


FIG. 14.

BATH FOR VOLATILITY TEST.

of 1 in. (25 mm.) above the surface of the lid. A suitable condenser shall be fitted to the larger to prevent the loss of toluene, and a thermometer to the smaller to control the production of the vapour.

The whole bath is set up on a wrought iron stand having legs of 10 in. (254 mm.) length, and the toluene boiled by means of gas jets placed under the bath.

The trough shall be filled with lead shot (0·064 in. (1·6 mm.) diameter), and the interior of the boiler or vapour jacket about half filled with commercially refined toluene.*

Beakers of glass, quartz or porcelain of 1½ in. (38 mm.) diameter by 1½ in. depth. In beakers of such dimensions the surface area of oil exposed is about 1·8 sq. in. (11·6 sq. cm.).

To avoid variation due to draughts of air passing over the beakers containing the tests, a movable screen, 6 in. deep by 12 in. by 7 in., should be provided and placed on the bath so as to surround the beakers.

METHOD.

Each beaker shall be weighed, 5 c.c. of oil pipetted into it and again weighed. The temperature of the bath having been brought up by boiling the toluene each beaker containing a sample shall be embedded right up to its lip in the lead shot in the trough and heated for eight hours. The beakers shall then be removed, cooled, and again weighed. The results shall be given in terms of loss of oil by weight expressed as a percentage of the original quantity.

* NOTE.—The boiling point of toluene at ordinary atmospheric pressure is 110° C. (230° F.), but the loss of heat in transmission makes the temperature of the oil under test only 100 C. (212° F.). The temperature of the oil should be checked by embedding one or more thermometers in the shot, or, if preferred, by putting a " blank " test in, with the thermometer placed in the oil.

CLASS VI.—TRANSFORMER AND SWITCH OILS.

SLUDGING VALUE.

I.P.T. Serial Designation—T.O. 21.
B.E.S.A. Specification—No. 148—1922.

OBJECTS.

To determine the approximate quantity of sludge formed by the oxidation of an oil when hot, and in presence of a metal, conditions which are met with in oil-filled electrical transformers and oil-filled electric switches.

The test is not recommended as a general test for oils other than transformer and switch oils.

APPARATUS (FIG. 15).

1. A round glass flask with bulb 3 in. (76 mm. diameter), with neck $2\frac{3}{4}$ in. (70 mm.) long, and of 1 in. (25 mm.) diameter.

A suitable oil-bath for immersion of the whole of the bulb of the flask shall be provided.

2. A glass condenser fitted to the flask, preferably by a ground-in joint. The upper portion of the condenser is provided with a one-hole rubber bung carrying a glass T-piece (B), which, in turn, carries the glass air inlet tube (A); the internal diameter of the T-piece (B) shall be 0·4 in. (10 mm.), and the internal diameter of the inlet tube (A) shall be 0·16 in. (4 mm.). The inlet tube (A) shall reach to within $\frac{1}{8}$ in. (3 mm.) of the bottom of the flask, and pass axially through the cylinder of copper.

3. Four 6 oz. wash-bottles with ground-in stoppers carrying inlet and exit tubes. For the experiment these are charged with—

(*a*) Liquid paraffin (*paraffin liquidum*).

(*b*) Sulphuric acid—concentrated.

(*c*) 10 per cent. solution of silver nitrate.

(*d*) Caustic soda solution—sp. gr. about 1·355 (32–33 per cent. NaOH).

In bottles *b*, *c*, *d*, the inlet tube shall dip $\frac{1}{2}$ in. (12·5 mm.) below the surface of the reagents.

4. A piece of pure sheet copper having a bright planished surface, and measuring 2 in. by 1¼ in. by 0·004 in. thick (51 mm. by 32 mm. by 0·01 mm.), shall be rolled into a cylinder 1¼ in. (32 mm.) high, so that the edges shall touch. The coiled strip shall stand vertically in the bottom of the flask.

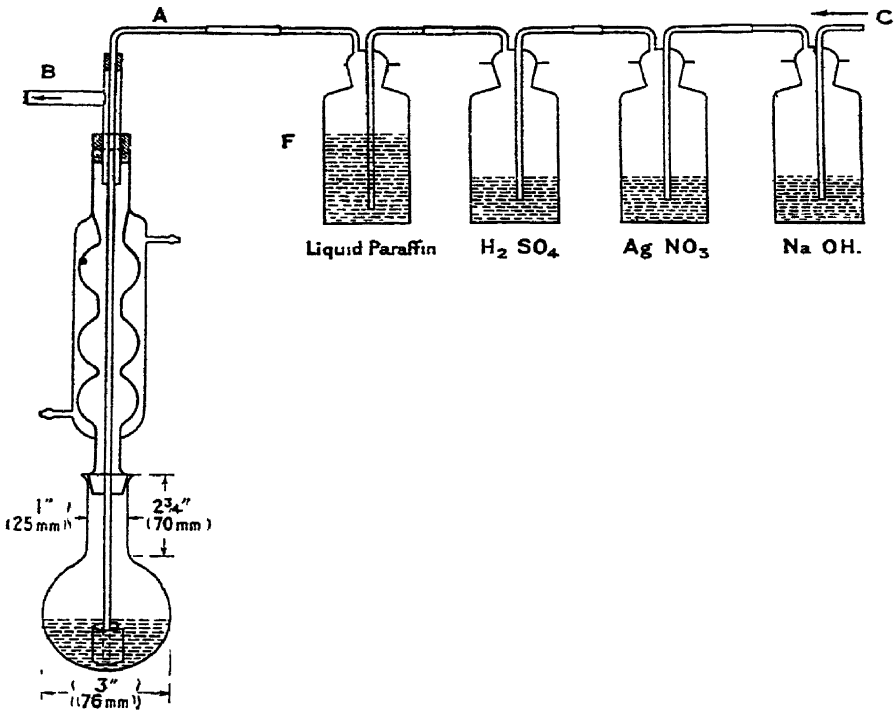


FIG. 15.

SLUDGING. VALVE APPARATUS.

5. The whole of the bulb of the flask shall be immersed in an oil bath, care being taken that, when the oil bath reaches 150° C. (302° F.), the ground-joint shall be above the surface of the oil, in order to ensure that no leakage of the latter into the test flash can occur.

6. A suitable blower which will force air through the bottles and contents of the flask at a rate of 0·07 cb. ft. (2·0 litres) per hour. The rate can be seen by the bubbles passing through the tell-tale bottle (*a*) containing the kerosene.

METHOD.

The temperature of the oil bath shall be brought to 150° C. (302° F.) and maintained at this temperature throughout the test. 100 c.c. of the oil shall be measured into the flask, the sheet-copper cylinder being placed around the air inlet tube so that it stands vertically.

The apparatus shall be connected up in the manner illustrated in Fig. 15, with the flask immersed in the oil bath, so that the ground joint is above the level of the oil in the bath. Air shall be bubbled through the apparatus, entering at C, at the rate of 0·07 cb. ft. (2·0 litres) per hour, during the whole period of the test.

After 45 hours' treatment, the flask shall be removed from the oil bath and allowed to cool below 100° C. (212° F.). The contents shall then be transferred to a beaker, diluted with three times its volume of petroleum spirit, and allowed to stand overnight for the sludge to separate out. Any deposit remaining in the flask shall be removed mechanically, and by washing with petroleum spirit if necessary, and added to the sludge in the beaker.

The petroleum spirit used shall be free from aromatic hydrocarbons and have a specific gravity of 0·70 to 0·72 at 15·5° C. (60° F.). Not less than 75 per cent. by volume shall distil over below 110° C. (230° F.), and the final boiling point shall not exceed 150° C. (302° F.).

The liquid shall then be decanted through a filter paper, and the deposit then washed on to the filter paper by means of petroleum spirit. The deposit on the filter paper shall then be thoroughly washed with petroleum spirit until free from oil and dried at 100° C. (212° F.) and dissolved in hot benzol and the solution collected in a tared flask. The solvent shall then be evaporated off on a water bath and the residue weighed.

In the event of the deposit being large, the bulk of it shall be detached from the filter paper, crushed and re-dried at 100° C. (212° F.) and then weighed. Any traces of deposit which adhere to the paper shall be removed by means of hot benzol and its weight obtained as above.

The result is expressed as a percentage, thus :—

$$\text{Percentage of sludge} = \frac{\text{Weight of deposit in grams.}}{\text{Specific gravity of the oil.}}$$

DIELECTRIC STRENGTH.

I.P.T. Serial Designation—T.O. 22.
B.E.S.A. Standard Specification—No. 148—1922.

APPARATUS (FIG. 16).

The test for dielectric strength (disruptive strength, breakdown

voltage) shall be carried out in a suitable oil vessel fitted with two electrodes arranged horizontally so that they are not less than 2 in. (5 cm.) below the surface of the oil in the vessel.

The electrodes shall be spheres of steel, brass or phosphor bronze, ½ in. (12·7 mm.) diameter. If the electrodes become pitted by the action of the spark they shall be renewed.

The spark gap between the electrodes shall be exactly 0·15 in. (3·81 mm.).

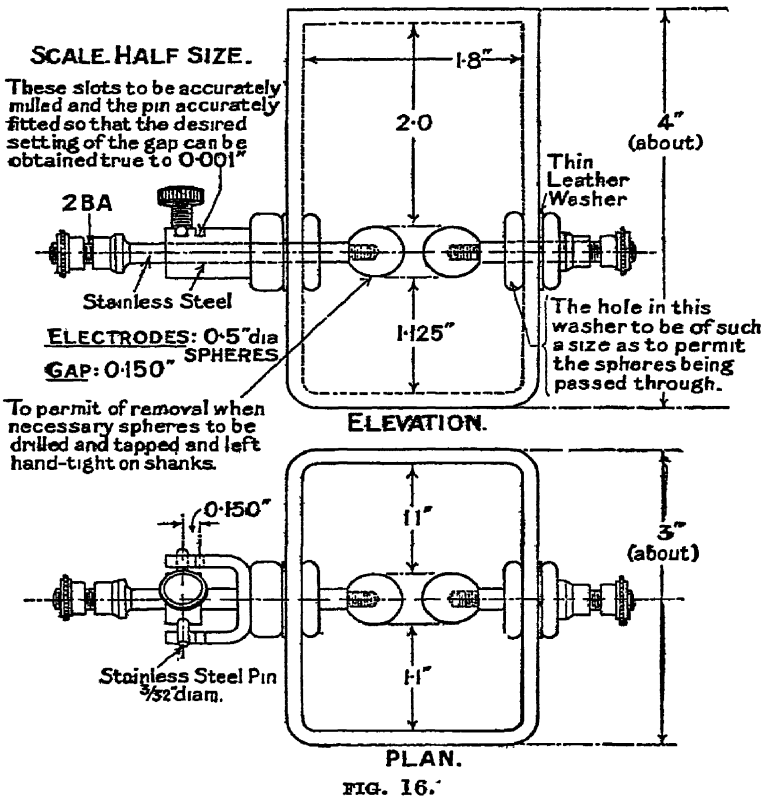


FIG. 16.

APPARATUS FOR DIELECTRIC STRENGTH.

PREPARATION OF THE APPARATUS.

The cell shall stand in a thick porcelain dish, or be otherwise suitably insulated, during tests.

The containing vessel and the electrodes shall be made absolutely clean and dry before carrying out the test. It is found advantageous

to carry out the final operation of removal of dust and fibres electrically as follows :—

The containing vessel should be filled with oil, and about half the test voltage applied to the electrodes for half a minute to attract dust and fibres. The electrodes should then be washed by pouring fresh oil over them, and the oil in the containing vessel poured away. This operation should be carried out four times, after which the container thus cleaned may be used for twenty consecutive tests without further cleaning.

METHOD OF TEST FOR DISRUPTIVE STRENGTH.

Before sampling, the barrel or other vessel shall stand for 24 hours with the bung or other opening at the top, and the samples shall be drawn from the bottom and thoroughly shaken before testing.

The oil used for this test shall not have been used for any other tests.

The volume of the oil used for each test shall not be less than 150 c.c. (roughly 5¼ fluid ounces). The temperature of the oil shall be between 15·5° C. and 20° C. (60° F. and 68° F.).

The test shall be commenced at a voltage of about one-third the test voltage, and shall be evenly increased until breakdown as rapidly as is consistent with its value being indicated by the measuring instrument.

Complete breakdown is known by the establishment of an arc.

Preliminary transient sparking which does not develop into an arc may be ignored.

VOLATILITY.

I.P.T. Serial Designation—T.O. 17.

The apparatus and method shall be as laid down in under L O. 17 (p. 61).

CLASS VII.—FUEL OILS

(OTHER THAN INTERMEDIATE OILS. Class IV.)

SPECIFIC GRAVITY.

I.P.T. Serial Designation—F.O. 1.

The apparatus and method shall be as laid down in the special directions for specific gravity (p. 1).

SULPHUR.

I.P.T. Serial Designation—F.O. 4.

The determination shall be made in the bomb calorimeter as described for Gas Oil (G.O. 4, p. 25).

ACIDITY.

I.P.T. Serial Designation—F.O. 5.

The inorganic acidity shall be determined by the method and the results expressed in the manner laid down under L.O. 5 (p. 43).

CALORIFIC VALUE.

I.P.T. Serial Designation—F.O. 6.

APPARATUS.

A suitable type of bomb calorimeter permitting the use of oxygen up to 30 atmospheres pressure shall be employed with thermometer, etc., in accordance with Method G.O. 6 (p. 26).

FLASH POINT.

I.P.T. Serial Designation—F.O. 7.
A.S.T.M. Serial Designation—D 93—21 T.

The I.P.T. Standard Pensky-Martens closed tester shall be used, as described under G.O. 7 (p. 28).

VISCOSITY.

I.P.T. Serial Designation—F.O. 8.

The Redwood Viscometer, No. I. or No. II., shall be used as laid down under L.O. 8 (p. 45).

CARBON RESIDUE.

I.P.T. Serial Designation—F.O. 9.
A.S.T.M. Serial Designation—D 47—21 T.

The apparatus and method shall be as laid down under G.O. 9 (p. 36).

ASH.

I.P.T. Serial Designation—F.O. 10.

The method shall be as laid down under G.O. 10 (p. 38).

COLD TEST.

I.P.T. Serial Designation—F.O. 11.

The apparatus and method shall be as laid down for the Pour Point Test, G.O. 11 (p. 38).

HARD ASPHALT.

I.P.T. Serial Designation—F.O. 12.

MATERIALS.

Petroleum Spirit.—To be freed from aromatic hydrocarbons by treatment with its own volume of 98—100 per cent. sulphuric acid. When tested by I.P.T. method G. 3 (p. 5) it shall distil between 60°—80° C.

METHOD.

10 grams of the sample shall be dissolved in 100 c.c. of petroleum spirit, thoroughly mixed and allowed to stand 24 hours. The whole shall be filtered through an 11 cm. folded filter and washed with petroleum spirit until the washings are colourless. The

material on the paper shall be dissolved in benzol, the solution being collected in a weighed conical flask. The solvent shall be distilled off on a water bath, the asphalt dried in a steam oven for 1 hour and weighed.

WATER.

I.P.T. Serial Designation—F.O. 14.
A.S.T.M. Serial Designation—D 95—23 T.

SAMPLE.

(1) The sample shall be thoroughly representative of the material to be tested and the portion of the sample used for the test shall be thoroughly representative of the sample itself. The difficulties in obtaining proper representative samples for this determination are unusually great so that the importance of sampling cannot be too strongly emphasised.

APPARATUS (FIG. 17).

(2) The form preferred is that of Dean and Stark, as shown in Fig. 17, and shall include the following :—

(*a*) Distillation flask, 500 c.c. capacity, of copper or well-annealed glass, round bottom.

(*b*) Reflux condenser, water cooled, glass-tube type.

(*c*) Special graduated distilling tube receiver of well-annealed glass graduated upward from 0 to 10 c.c. in 0·1 c.c. divisions, accurate to 0·05 c.c., (Fig. 17).

(*d*) Gas burner or suitable electric heater.

(3) An optional form may be used and shall include the following :—

(*a*) Distillation flask, with side tube ; 500 c.c. capacity.

(*b*) Condenser, water cooled, glass-tube type, free from traps retarding complete draining.

(*c*) Receiver, of glass, capacity about 125 c.c., I.P.T water and sediment tube Fig. 18 (p. 74), or equivalent.

(*d*) Gas burner or suitable electric heater.

(*e*) Thermometer, engraved-stem type, to indicate 400° F.

(4) The diluent used in this method shall be gasoline, free from water. When subjected to distillation (I.P.T. Method G 3) it shall show 5 per cent. at a temperature not above 212° F. (100° C.) nor below 194° F. (90° C). It shall show 90 per cent. at a temperature not above 410° F. (205° C.).

METHOD.

(5) Exactly 100 c.c. of the oil to be tested shall be measured in an accurate 100 c.c. graduated cylinder at room temperature and poured into the distillation flask. The oil adhering to the walls of the 100 c.c. graduated cylinder shall be transferred to the

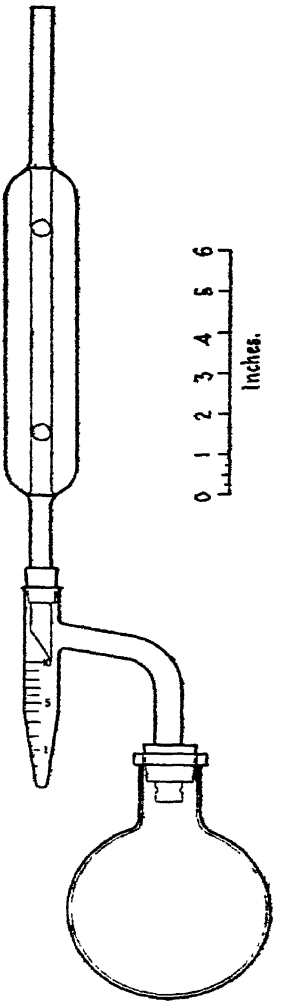


FIG. 17.

DETERMINATION OF WATER.

distillation flask by rinsing with two successive 50 c.c. portions of gasoline, the cylinder being allowed to drain each time. The oil and gasoline in the distillation flask shall be thoroughly mixed by swirling the flask with proper care to avoid any loss of material. A boiling stone, such as a piece of unglazed porcelain, may be introduced for the purpose of preventing bumping during the subsequent distillation.

(6) The apparatus shall be assembled in the following manner :—

(*a*) If the preferred form of apparatus is used, the distillation flask shall be connected with the special graduated distilling tube receiver and this receiver with the reflux condenser by means of tight-fitting corks as shown in Fig. 17.

(*b*) If the optional form of apparatus is used, the vapour outlet tube of the distillation flask shall be inserted into the condenser, a tight connection being made by means of a cork. The receiver shall be supported in a suitable position at the other end of the condenser without the use of a cork or other connection. Proper precautions shall be taken to prevent the introduction into the receiver of water from any source other than the subsequent distillation process.

The mouth of the distillation flask shall be closed by a tight-fitting cork supporting the thermometer in such a position that the top of the thermometer bulb is level with the bottom of the vapour outlet tube, where the latter is joined to the neck of the flask.

(7) Either form of apparatus being assembled as prescribed, heat shall be applied to the distillation flask and shall be so regulated that the condensed distillate falls from the end of the condenser at the rate of 2 to 4 drops per second.

(8)—(*a*) With the preferred form of apparatus, distillation shall be continued at the specified rate until no water is visible on any part of the apparatus except at the bottom of the distilling tube receiver. This operation usually requires less than one hour. A persistent ring of condensed water in the condenser tube shall be removed by increasing the rate of distillation for a few minutes.

(*b*) With the optional form of apparatus, distillation shall be continued at the specified rate until the thermometer indicates a vapour temperature of 400° F. and until all condensed water has disappeared from the walls of the distillation flask and of the condenser.

(9) With either form of apparatus, the volume of condensed water, measured in the graduated receiver at room temperature, shall be recorded as " — per cent Water, I.P.T. Method."

ACCURACY.

(10) With proper care and attention to details, duplicate determinations of water by this method should not differ from each other by more than 0·2 c.c. of water provided the graduated receiver is accurate and readable to this degree.

When the sample to be tested contains more than 10 per cent. of water, the volume of material used shall be decreased to that which will yield somewhat less than 10 c.c. of water. Otherwise the procedure shall be conducted as prescribed.

WATER AND SEDIMENT.

I.P.T. Serial Designation—F.O. 14a.
A.S.T.M. Serial Designation—D 96—21 T.

1. This method may be used for crude mineral oils and fuel oils. A centrifuge method for "Water and Sediment" is not entirely satisfactory because the amount of water obtained is nearly always lower than the actual water content. Nevertheless, on account of the wide use of the centrifuge for this purpose, it is desirable that the method of making the determination be standardised as far as possible. It must be clearly understood that the reading of the centrifuge tube includes both the sediment and the precipitated water. Accurate determination of water content if desired shall be made by the preceding method.

2. The sample shall be thoroughly representative of the material in question and the portion used for the test shall be thoroughly representative of the sample itself. The difficulties in obtaining proper representative samples for this determination are unusually great so that the importance of sampling cannot be too strongly emphasised.

APPARATUS (FIG. 18).

3. The centrifuge shall be capable of whirling at the required speed at least two 100-c.c. centrifuge tubes filled with water. It shall be of sound design and rugged construction so that it may be operated without danger. The tube carriers shall be so designed that the glass centrifuge tubes may be cushioned with water, rubber or other suitable material. The tube holders shall be surrounded during the operation by a suitable metal shield or case, strong enough to eliminate danger if any breakage occurs.

The preferred form of centrifuge shall have a diameter of swing (tip to tip of whirling tubes) of 15 to 17 in. and a speed of at least 1500 r.p.m. or the equivalent. If the available centrifuge has a diameter of swing varying from these limits, it shall be run at the proper speed to give the same centrifugal force at the tips

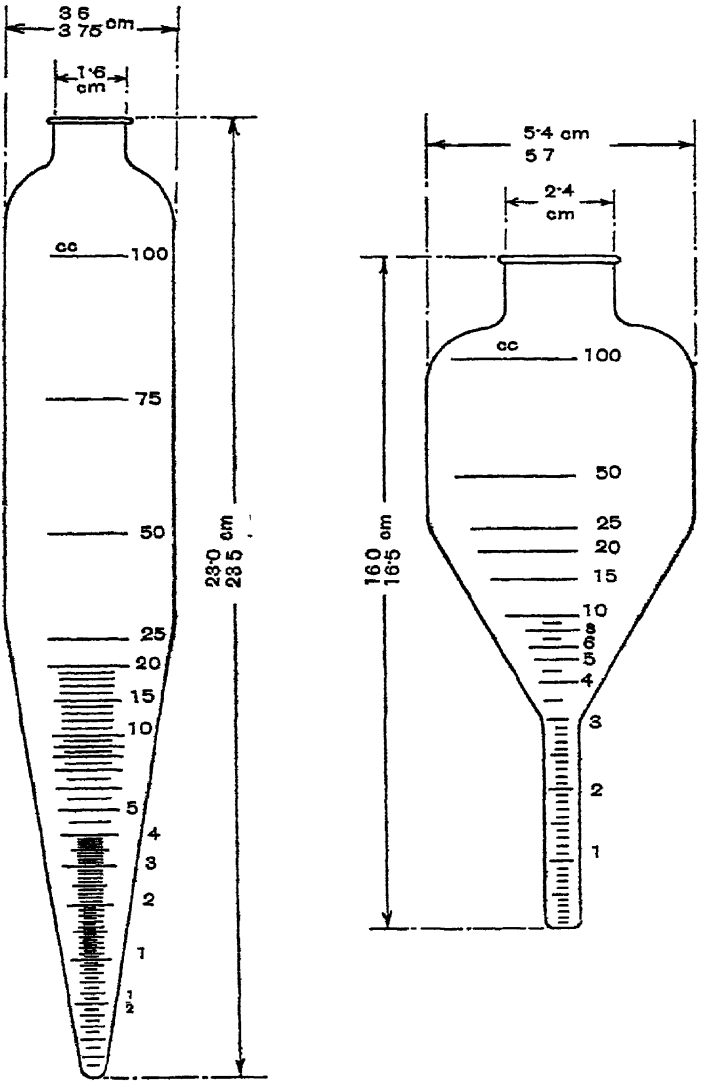


FIG. 18.

CENTRIFUGE TUBES FOR WATER AND SEDIMENT.

of the tubes as that obtained with the preferred form of centrifuge. The proper speed shall be calculated from the following formula in which d represents diameter of swing (tip to tip of whirling tubes) of the centrifuge used :—

$$\text{R.p.m.} = 1500\sqrt{\frac{16}{d}}$$

4. The centrifuge tubes shall be made of suitable glass and thoroughly annealed. The total capacity shall be about 125 c.c. and the mouth shall be suitably constricted for closing with a cork. The graduations shall be clear and distinct, reading upward from the bottom of the tube in accordance with Fig. 18 and with the following limits of error :—

Range. c.c.	Limit of Error. c.c.
0 to 3	± 0·05
3 to 5	± 0·2
5 to 10	± 0·5
At 50	± 1·0
At 100	± 1·0

The shape is optional provided it does not conflict with the other requirements.

5. The water or oil bath shall be of sufficient depth for immersing the centrifuge tubes in a vertical position to the 100-c.c. mark. Means shall be provided for heating this bath to 100° F.

METHOD.

6. (*a*) Exactly 50 c.c. of 90 per cent. benzol shall be measured into each of two centrifuge tubes and exactly 50 c.c. of the oil to be tested shall then be added to each. The centrifuge tubes shall be tightly stoppered and shall be shaken vigorously until the contents are thoroughly mixed. The temperature of the bath shall be maintained at 100° F. and the centrifuge tubes shall be immersed therein to the 100 c.c. mark for 10 minutes.

(*b*) The two centrifuge tubes shall then be placed in the centrifuge on opposite sides and shall be whirled at a rate of 1400 to 1500 r.p.m., or the equivalent, for 10 minutes. The combined volume of water and sediment at the bottom of each tube shall be read and recorded, estimating to 0·1 c.c. if necessary. The centrifuge tubes shall then be replaced in the centrifuge, again whirled for 10 minutes as before and removed for reading the volume of water and sediment as before. This operation shall be repeated until the combined volume of water and sediment in each tube remains constant for three consecutive readings. In general, not more than four whirlings will be required.

7. The combined total volume of water and sediment shall be read on each tube, estimating to 0·1 c.c. if necessary. The sum of the two readings shall be recorded as " percentage of water and sediment, I.P.T. centrifuge method."

ACCURACY.

8. With care and proper attention to details, duplicate determinations of water and sediment by this method should not differ by more than 0·2 c.c. provided the centrifuge tubes are accurate and readable to this degree.

CLASS VIII.—ASPHALTS

SPECIFIC GRAVITY.

I.P.T. Serial Designation—A. 1.

Solid Materials.—Where applicable the method of displacement of water by a known weight of material shall be employed.

Other Materials.—For semi-solid materials the specific gravity shall be determined in a graduated flask of not less than 100 c.c. capacity, or a special pyknometer, the material being heated to promote fluidity. The contents of the flask may be cooled to 60° F. if their nature permits and the volume to the graduation mark adjusted with a little more of the melted material. Where this cooling is not practicable the specific gravity shall be determined at higher temperatures, the material being kept fluid, and the proper temperature correction made. (See pp. x. and 1.)

SULPHUR.

I.P.T. Serial Designation—A. 4.

The determination shall be made in a bomb calorimeter by the method laid down under G.O. 4 (p. 25) with such modifications of the preparation of the sample for combustion in the bomb as its nature requires.

FLASH POINT AND FIRE POINT (OPEN).

I.P.T. Serial Designation—A. 7a.

The apparatus shall be the Pensky-Marten cup (without cover) and method as laid down under L O. 7a (p. 44).

MOBILITY.

I.P.T. Serial Designation—A. 8.

This shall be determined at suitable temperatures in the Redwood No. II. Viscometer (arranged to permit of temperatures

up to 420° F. being obtained), as laid down under L.O. 8 (p. 50). The following temperatures are recommended :—200°, 300°, 400° F., and for flux oils 200° F.

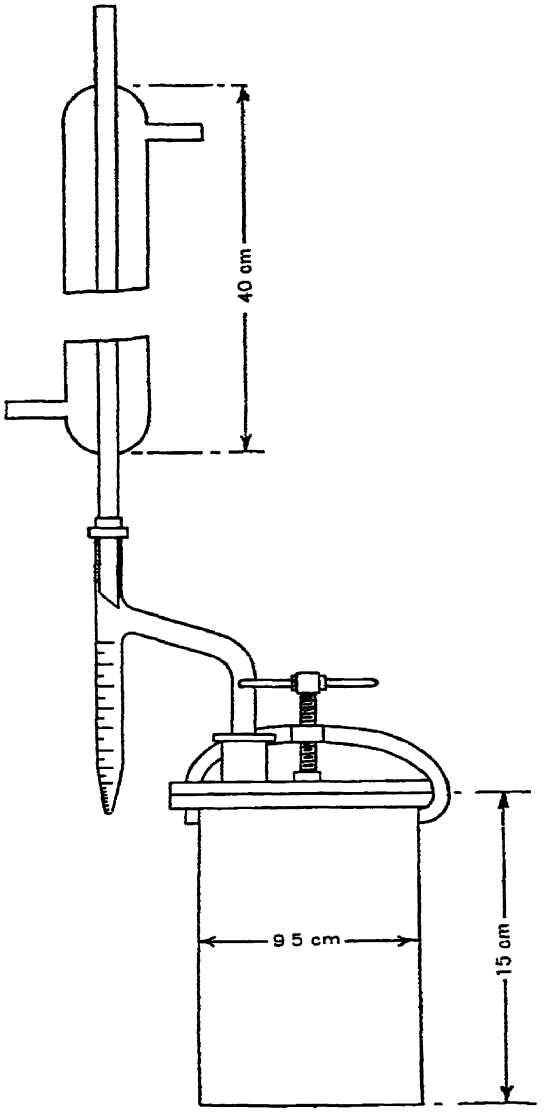


FIG. 19.

WATER IN ASPHALTIC SUBSTANCES.

CARBON RESIDUE.

I.P.T. Serial Designation—A. 9.
A.S.T.M. Serial Designation—D 47—21 T.

The method shall be as laid down under G.O. 9 (p. 36).

ASH.

I.P.T. Serial Designation—A. 10.

A suitable quantity of the sample shall be slowly carbonised in an open platinum dish and the residue incinerated until all carbon has been burnt off.

WATER.

I.P.T. Serial Designation—A. 14.
A.S.T.M. Serial Designation—D 95—21 T.

The apparatus and method shall, in general, be that laid down under F.O. 14 (p. 70), but for asphaltic substances it is generally desirable to use a metal still, preferably of copper, Fig. 19, having a faced flange at the top of which the head is tightly attached by means of a clamp. The head is also of metal, brass or copper, and is provided with a tubulation of one inch diameter.

A heavy paper gasket moistened with the diluent shall be inserted between the lid and flange before attaching the clamp.

The burner used with the metal still shall be a ring gas burner of 4 in. (100 mm.) inside diameter.

The diluent used for bituminous materials derived from petroleum shall conform to that laid down under F.O. 14.

For bituminous materials derived from coal tar, water-gas, tar, etc., a coal tar naphtha shall be employed. When tested by the I.P.T. Standard Distillation Test (G. 3, p. 5) 98 per cent. shall distil between 120° C. (248° F.) and 250° C. (482° F.).

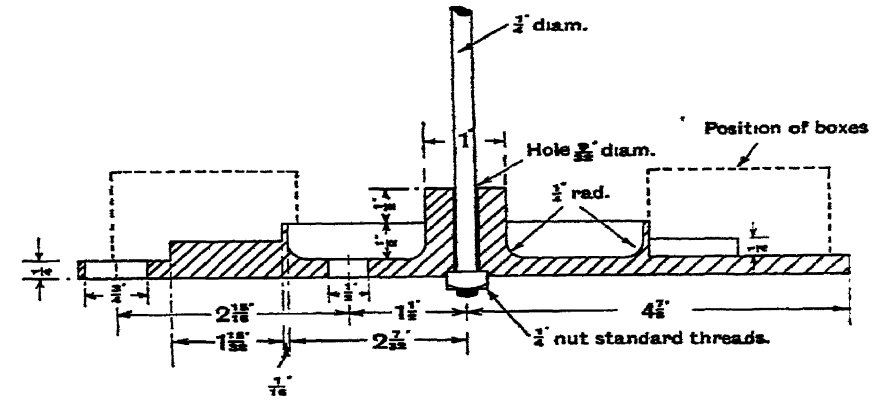
LOSS ON HEATING.

I.P.T. Serial Designation—A 17.
A.S.T.M. Serial Designation—D 6—20.

1. This test is used to determine the loss in weight (exclusive of water) of oil and asphaltic compounds when heated as hereinafter prescribed. The material under examination must therefore first be tested for water, and if water is found to be present it must be removed by suitable methods of dehydration before the material is subjected to the loss on heating test; or another sample obtained which is free from water.

APPARATUS (FIG. 20).

2. The oven may be either circular or rectangular in form and may be heated by either gas or electricity. Its interior dimensions shall be as follows :—Height, not less than 40·5 cm.



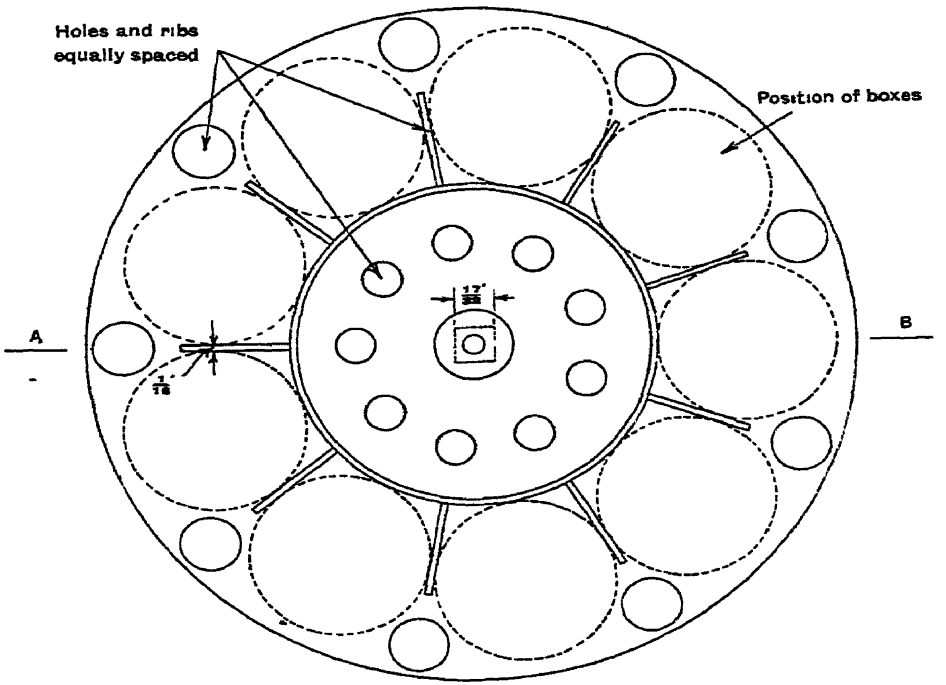


FIG. 20.

PLAN.

ALUMINIUM SHELF FOR ASPHALT SAMPLES.

(16 in.); width and depth or diameter, at least 4 cm. ($1\frac{5}{8}$ in.) greater than the diameter of the revolving shelf.

It shall be well ventilated and shall be fitted with a window in the upper half of the door, so placed and of sufficient size to permit the accurate reading of the thermometer without opening the door. It shall also be provided with a perforated circular shelf preferably of approximately 25 cm. ($9\frac{3}{4}$ in.) in diameter. This shelf shall be placed in the centre of the oven and shall be suspended by a vertical shaft and provided with mechanical means for rotating it at the rate of 5 to 6 revolutions per minute. It shall be provided with recesses equidistant from the central shaft in which the tins containing the samples are to be placed. (A recommended form of aluminium shelf is shown in Fig. 20).

THERMOMETER SPECIFICATION.

Type.—Mercury in glass. Solid stem. Nitrogen filled.

Stem.—Lead glass or other suitable glass. Enamel back.

Bulb.—Cylindrical. Powell's normal glass, Jena 16,[111] or other approved glass. Length 10 to 15 mm.

Range.—150° to 175° C.

Immersion.—Total.

Dimensions.—Overall length, 130 to 150 mm. Distance from bottom of bulb to first graduation mark approximately 50–55 mm.

Expansion Chamber.—Required.

Finish.—Glass ring.

Graduation.—Every 0·5 degree. Each degree larger than the ½ degree and each 5° and 10° shall be indicated by larger lines than the degrees. The 0·5 degree division shall be approximately 1·5 mm.

Figuring.—The scale shall be figured at each 5°.

Marking.—"I.P.T. Asphalt. A.17." Identification number. Maker's (or vendor's) name or trade mark.

Each thermometer shall be tested at the National Physical Laboratory, where satisfactory thermometers will be marked with the N.P.L. monogram and year of test, and a certificate of corrections issued.

4. The container in which the sample is to be tested shall be of tin or aluminium, cylindrical in shape, and shall have a flat bottom. Its inside dimensions shall be substantially as follows:—Diameter 55 mm. (2·2 in.), depth 35 mm. (1·4 in.).

PREPARATION OF SAMPLE.

5. The sample as received shall be thoroughly stirred and agitated, warming, if necessary, to insure a complete mixture before the portion for analysis is removed.

METHOD.

6. 50 g. of the water-free material shall be weighed into a tared container conforming to the requirements of §4, p. 80. The oven shall be heated to a temperature of 163° C. (325° F.), and the tin box containing the sample placed in one of the recesses of the revolving shelf. The thermometer shall be immersed for the depth of its bulb in a separate 50-gram sample of the material under test, placed in a similar container, and shall be conveniently suspended from the vertical shaft. This sample shall rest in one of the recesses upon the same shelf and revolve with the sample or samples under test. The oven shall be closed and the shelf rotated 5 to 6 revolutions per minute during the entire test. The temperature shall be maintained at 163° C. (325° F.) for 5 hours, then the sample shall be removed from the oven, cooled and weighed, and the loss due to volatilization calculated.

7. During the 5-hour period the temperature shall not vary more than 1° C. All tests showing a greater variation in temperature shall be rejected.

ACCURACY.

8. Up to 5 per cent. loss in weight the results obtained may be considered as correct within 0·5. Above 5 per cent. loss in weight the numerical limit of error increases 0·01 for every 0·5 per cent. increase in loss by volatilization as follows :—

Volatilization Loss, per cent.	Numerical Correction.	True Volatilization Loss, per cent.
5·0 ..	±0 5 ..	4·5 to 5·5
5·5 ..	±0·5 ..	5·0 ,, 6·0
6·0 ..	±0·5 ..	5·5 ,, 6·5
10·0 ..	±0·6 .	9 4 ,, 10·6
15·0 ..	±0·7 ..	14·3 ,, 15·7
25·0 ..	±0·9 ..	24·1 ,, 25·9
40·0 ..	±1·2 ..	38 8 ,, 41·2

PRECAUTIONS.

9. Under ordinary circumstances a number of samples having about the same degree of volatility may be tested at the same time. Samples varying greatly in volatility should be tested separately. Where extreme accuracy is required not more than one material should be tested at one time and duplicate samples of it should be placed simultaneously in the oven. Such duplicates shall check within the limits of accuracy given above. Results obtained on samples showing evidences of foaming during the test shall be rejected.

NOTE.—If additional periods of heating are desired, it is recommended that they be made in successive increments of 5 hours each.

When the penetration of the sample after heating is required, the residue in the container shall be melted at the lowest possible temperature and thoroughly mixed by stirring, taking care to avoid incorporating air bubbles in the mass. It shall then be brought to the standard temperature and tested as prescribed under the directions for the Standard Test for Penetration of Bituminous Materials.

PENETRATION.

I.P.T. Serial Designation—A. 18.
A.S.T.M. Serial Designation—D 5—21.

DEFINITION.

1. Penetration is defined as the consistency of a bituminous material, expressed as the distance that a standard needle vertically penetrates a sample of the material under known conditions of loading, time and temperature. Where the conditions of test are not specifically mentioned, the load, time and temperature are understood to be 100 grams, 5 seconds, 25° C. (77° F.), respectively, and the units of penetration to indicate hundredths of a centimetre.

APPARATUS.

2. The container for holding the material to be tested shall be a flat-bottom, cylindrical dish, 55 mm. ($2\frac{3}{16}$ in.) in diameter and 35 mm. ($1\frac{3}{8}$ in.) deep.

The needle for this test (Fig. 21) shall be a cylindrical steel rod 50·8 mm. (2 in.) long, having a diameter of 1·01 to 1·02 mm. and having a taper of 6·34 to 6·36 mm. measured on the axis. After tapering, the point shall be " blunted ' by grinding off to a truncated cone, the smaller base of which shall be from 0·14 to 0·16 mm. in diameter. The penetration needle shall be gauged for dimensions by the National Physical Laboratory.

The water bath shall be maintained at a temperature not varying more than 0·1° C. from 25° C. (77° F.). The volume of water shall be not less than 10 litres and the sample shall be immersed to a depth of not less than 10 cm. (4 in.) and shall be supported on a perforated shelf not less than 5 cm. (2 in.) from the bottom of the bath.

Any apparatus which will allow the needle to penetrate without appreciable friction, and which is accurately calibrated to yield results in accordance with the definition of penetration, will be acceptable.

The transfer dish for container shall be a small dish or tray of such capacity as will insure complete immersion of the container during the test. It shall be provided with some means which will insure a firm bearing and prevent rocking of the container.

PREPARATION OF THE SAMPLE.

3. The sample shall be completely melted at the lowest possible temperature and stirred thoroughly until it is homogeneous and free from air bubbles. It shall then be poured into the sample container to a depth of not less than 15 mm. ($\frac{5}{8}$ in.). The sample shall be protected from dust and allowed to cool in an atmosphere not lower than 18° C. (65° F.) for one hour. It shall then be placed in the water-bath along with the transfer dish and allowed to remain one hour.

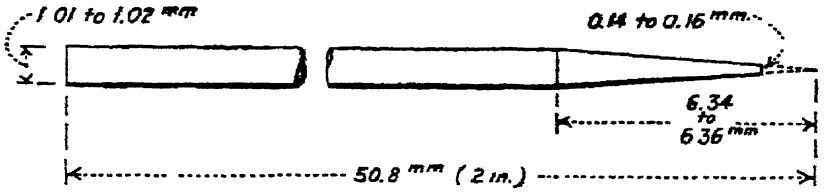


FIG. 21.

NEEDLE FOR PENETRATION TEST.

METHOD.

4. (*a*) In making the test the sample shall be placed in the transfer dish filled with water from the water-bath of sufficient depth to completely cover the container. The transfer dish containing the sample shall then be placed upon the stand of the penetration machine. The needle, loaded with specified weight, shall be adjusted to make contact with the surface of the sample. This may be accomplished by making contact of the actual needle point with its image reflected by the surface of the sample from a properly placed source of light. Either the reading of the dial shall then be noted or the needle brought to zero. The needle is then released for the specified period of time, after which the penetration machine is adjusted to measure the distance penetrated. At least three tests shall be made at points on the surface of the sample not less than 1 cm. ($\frac{3}{8}$ in.) from the side of the container and not less than 1 cm. ($\frac{3}{8}$ in.) apart. After each test the sample and transfer dish shall be returned to the water-bath and the needle shall be carefully wiped towards its point with a clean, dry cloth to remove all adhering bitumen. The reported penetration shall not differ more than four points between maximum and minimum.

(*b*) When desirable to vary the temperature, time and weight, and in order to provide for a uniform method of reporting results when variations are made, the samples shall be melted and cooled in air as above directed. They shall then be immersed in water or brine, as the case may require, for one hour at the temperature desired. The following combinations are suggested :—

At 0° C. (32° F.) 200 gram weight, 60 seconds.
At 46° C. (115° F.) 50 gram weight, 5 seconds.

DUCTILITY.

I.P.T. Serial Designation—A. 19.

DEFINITION.

Ductility is a test of the consistency of bituminous material, the results of which are expressed in the distance in centimetres that a block of standard shape and dimensions and at a standard temperature can be drawn out before fracture.

APPARATUS.

The standard pattern Dow ductility machine shall be used, with the following modification :—The distance between the carrier plate and the bottom of the trough shall be 1 cm.

It is further recommended that :—

(1) The standard instrument shall be mechanically driven, preferably by electrical means.

(2) A milk glass sheet be placed at the bottom of the trough, so as to present an even surface which will not break the thread of bitumen under test.

(3) The moulds have sloping side pieces, so that a slight inward pressure binds the whole mould together.

(4) Hot water from a suitable supply or electric heating be used instead of the present practice of heating by gas jets placed beneath the trough.

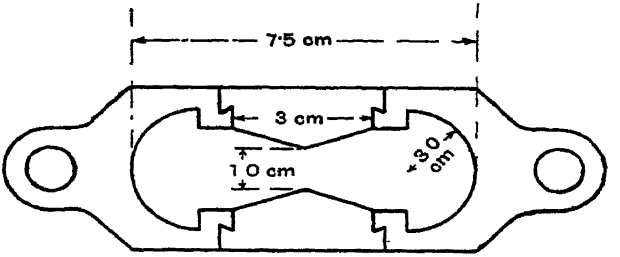


FIG. 22.
MOULD FOR DUCTILITY TEST.

The dimensions of the mould shall be approximately :—

	cm.
Internal length	7·5
Distance between ends of clips	3·0
Extreme width	3·0
Internal width, at mouth of clips	2·0
Thickness of sample block..	1·0

CONDITIONS FOR TEST.

The water-bath for the samples must be maintained at all depths at a temperature of 15° C. (60° F.), constant to within 0·1° C. The volume of water shall not be less than 10 litres, and the sample shall be immersed to a depth of not less than 10 cm. (4 in.), and shall be supported on a perforated shelf not less than 5 cm. (2 in.) from the bottom of the bath.

PREPARATION OF THE SAMPLE.

A clean representative sample shall be heated, with occasional stirring, in a round-bottom nickel vessel. The quantity must be sufficient for a test in triplicate. Place the dish well above a source of heat, and raise the temperature at such a rate that 130° C. (266° F.) is reached in one hour. If bubbles or froth persist, heat at this temperature for another half-hour. If they are still present, they shall be broken with a tiny gas flame ; but if they are not to be driven off, another sample shall be taken for testing.

If the sample is markedly impure, it shall be heated for as short a time as is possible without the risk of over-heating at 150° C (302° F.) and strained through a 50-mesh sieve.

Precautions.—It is essential that all heating of the bitumen shall be at as low a temperature and as slow a rate as is practicable, and at the same time in accordance with the above instructions.

The base-plate and the side pieces of three brass moulds shall be amalgamated with mercury. The parts of the moulds shall be assembled on their plates and the melted sample poured almost to overflowing. A warmed strip of amalgamated brass plate shall be pressed over the central portion to level the sample, and the ends levelled with a warmed sharp knife only.

The poured samples shall cool for one hour in air, protected from dust, and for one and a half hours in water at the temperature of the test.

METHOD.

The temperature of the water in the trough of the ductility machine shall be raised to that desired, normally 15·5° C., and the heating arrangements adjusted so that it remains constant. The side pieces of the moulds shall be removed and the samples transferred from their base plates to their positions in the machine. The carriage shall be set in motion at the standard speed of 5 cm. per minute, and the distance travelled when each thread breaks noted. The highest of these figures shall be reported.

When it is required to examine more broadly the general characteristics of a sample, tests may be made at other temperatures, those suggested being 20° C. and 25° C.

Tolerance.—5 per cent.

SOFTENING POINT
(OTHER THAN TAR PRODUCTS).

(RING-AND-BALL METHOD.)

I.P.T. Serial Designation—A. 20.
A.S.T.M. Serial Designation—D 36—21.

DEFINITION.

1. The softening of bituminous materials generally takes place at no definite moment or temperature. As the temperature rises, they gradually and imperceptibly change from a brittle or exceedingly thick and slow flowing material to a softer and less viscous liquid. For this reason the determination of the softening point must be made by a fixed, arbitrary and closely defined method if the results obtained are to be comparable.

APPARATUS (FIG. 23).

2. The apparatus shall consist of the following :—

(A) A brass ring 15·875 mm. ($\frac{5}{8}$ in.) in inside diameter and 6·35 mm. ($\frac{1}{4}$ in.) deep; thickness of wall, 2·38 mm. ($\frac{3}{32}$ in.); permissible variation on inside diameter and thickness of ring, ±0·25 mm. (0·01 in.). This ring shall be attached in a convenient manner to a No. 15 S.W.G. brass wire, diameter 1·83 mm. (0·072 in.). See Fig. 23.

(B) A steel ball 9·53 mm. ($\frac{3}{8}$ in.) in diameter weighing between 3·45 and 3·55 grams. A ball-bearing ball invariably conforms with the requirements.

(C) A glass vessel, capable of being heated, not less than 8·5 cm. (3·3 in.) in diameter and measuring 10·5 cm. (4 in). in depth from the bottom of the flare. (A 600 c.c. beaker, low form, meets this requirement.)

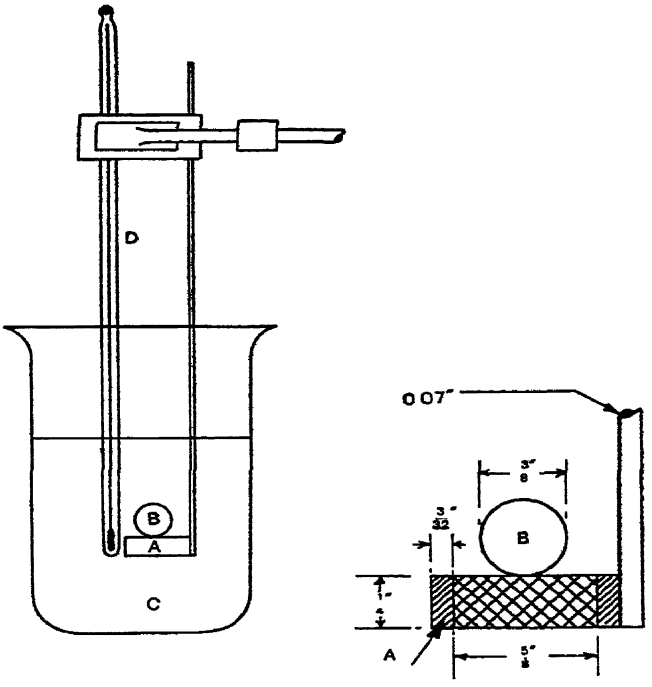


FIG. 23.

RING AND BALL APPARATUS FOR SOFTENING POINT.

THERMOMETER SPECIFICATIONS.

Type —Mercury in glass. Solid stem.

Stem —Lead or other suitable glass. Enamel back. Diameter 6·5 to 7·5 mm.

Bulb.—Cylindrical. Powell's normal glass. Jena 16,^{111} or other approved glass. Length, not over 14 mm. Diameter,. 4·5 to 5·5 mm.

Range.—0° to 80° C. (32 to 176° F.).

Immersion.—Total.

Dimensions.—Total length, 370–400 mm. The distance from the bottom of the bulb to the first scale division shall not be less than 75 mm. The distance from the 0° mark to the 80° mark shall be between 230 and 275 mm.

Expansion chamber.—Required.

Finish.—Glass ring.

Graduation.—0·5° division for C. or F. scale. Each degree longer than the ½ degrees, and each 5° and 10° shall be indicated by longer lines than the degrees.

Figuring.—The scale shall be figured at each 5° or 10°.

Marking.—"I.P.T. A20." Identification number. Maker's (or vendor's) name or trade mark.

Each thermometer shall be tested at the National Physical Laboratory, where satisfactory thermometers will be marked with the N.P.L. monogram, and year of test, and a certificate of corrections issued.

PREPARATION OF SAMPLE.

3. The sample shall be melted and stirred thoroughly, avoiding incorporating air bubbles in the mass, and then poured into the ring so as to leave an excess on cooling. The ring, while being filled, should rest on a brass plate which has been amalgamated to prevent the bituminous material from adhering to it. After cooling, the excess material shall be cut off cleanly with a slightly heated knife.

METHOD.

(*A*) *Bituminous Materials having Softening Points* 80° *C.* (176° *F.*) *or below.*

4. The apparatus shall be assembled as shown in Fig. 23, and the glass vessel filled to a depth of 3 to 3½ in., with freshly boiled, distilled water at 5° C. The ball shall be placed in the centre of the upper surface of the bitumen in the ring, and the ring suspended in the water so that the lower surface of the filled ring is exactly 1 inch above the bottom of the glass vessel and its upper surface is 2 inches below the surface of the water. It shall remain in the water for 15 minutes before applying heat. The thermometer shall be suspended so that the bottom of the bulb is level with the bottom of the ring and within ¼ inch, but not touching the ring.

5. Heat shall be applied in such a manner that the temperature of the water is raised 5° C. (9° F.) each minute.

6. The temperature recorded by the thermometer at the instant the bituminous material touches the bottom of the glass vessel shall be reported as the softening point.

7. The rate of rise of temperature shall be uniform and shall not be averaged over the period of the test. The maximum permissible variation for any minute period after the first three shall be ±0°·5 C. (0°·9 F.). All tests in which the rate of rise in temperature exceeds these limits shall be rejected.

(*B*) *Bituminous Materials having Softening Points above 80° C. (176° F.).*

8. The same method as given under (*A*) shall be used, except that glycerin shall be used instead of water, that the initial temperature shall be 30° C., and that the thermometer shall conform to the following specifications :—

THERMOMETER SPECIFICATIONS.

Type.—Mercury in glass. Solid stem. Nitrogen filled.

Stem.—Lead or other suitable glass. Enamel back. Diameter 6·5 to 7·5 mm.

Bulb.—Cylindrical. Powell's normal glass, Jena 16,[111] or other approved glass. Length not over 14 mm. Diameter, 4·5–5·5 mm.

Range.—30° to 160° C.

Immersion.—Total.

Dimensions.—Total length, 370 to 400 mm. Distance from bottom of the bulb to 30° mark not less than 75 mm. Distance from 30° mark to 160° mark shall be between 230 and 275 mm.

Expansion Chamber.—Required.

Finish.—Glass ring.

Graduation.—0·5° divisions. Each degree larger than the ½ degrees, and each 5° and 10° shall be indicated by longer lines than the degrees.

Figuring.—The scale shall be figured at each 10°.

Marking.—" I.P.T. A20." Identification number. Maker's (or vendor's) name or trade mark.

Each thermometer shall be tested at the National Physical Laboratory, where satisfactory thermometers will be marked with the N.P.L. monogram and year of test, and a certificate of corrections issued.

PRECAUTIONS.

9. The use of freshly boiled distilled water is essential, as otherwise air bubbles may form on the specimen and affect the accuracy of the results. Rigid adherence to the prescribed rate of heating is absolutely essential in order to secure accuracy of results.

A sheet of paper placed on the bottom of the glass vessel and conveniently weighted will prevent the bituminous material from sticking to the glass vessel, thereby saving considerable time and trouble in cleaning.

ACCURACY.

10. The limit of accuracy of the test is $\pm 0°{\cdot}5$ C. ($0°{\cdot}9$ F.).

WAX IN ASPHALT.

I.P.T. Serial Designation—A. 31.

METHOD.

From 1 to 3 grams of the sample (according to the amount of suspected wax) shall be weighed into an evaporating basin of about 200 c.c. capacity; approximately 10 times the weight of pure concentrated sulphuric acid added, and the basin and contents warmed on a sand bath.

When the bitumen shows signs of melting the mixture shall be stirred at intervals. Frothing occurs and sulphur dioxide is given off, and the bitumen assumes a porous spongy carbonaceous mass. This should occur in from 15 to 20 mins. and overheating shall be avoided.

The basin and contents shall be allowed to cool. The bitumen should now have the appearance of a dry soft coke.

The carbonaceous mass shall be broken up roughly with a glass rod and 50 to 100 c.c. of water added and sufficient solid sodium carbonate to make the whole distinct alkaline.

The contents shall be filtered through a Buchner funnel and the charred mass washed once or twice with water.

The bulk of the char shall be removed and roughly crushed in a mortar, and the crushed mass, the filter paper and the small piece of cotton wool used to wipe out the mortar transferred to a flask (a 200 c.c. conical for preference) and the flask fitted with a reflux condenser.

50 c.c. of a mixture of ethyl ether and alcohol, equal parts, shall be poured through the condenser, and the mixture allowed to boil gently for 15 mins.

The contents whilst still hot shall be poured through a funnel containing a pleget of cotton-wool into a beaker standing in a basin of iced water.

The extraction shall be repeated twice more with fresh quantities of ether/alcohol, filtering these extracts whilst hot through the same cotton-wool into the same beaker.

The wax rapidly separates out in flakes, the beaker being finally cooled by standing it in ice/salt mixture.

A Gooch crucible shall be prepared with asbestos in the usual way, dried and weighed and attached to the filter pump. Pour through the Gooch a few c.cs. of alcohol/ether which has been cooled in ice/salt, then, with the vacuum slightly on, the cold wax suspension shall be added. The large bulk of cold alcohol/ether prevents undue heating during the short time of filtering.

The mass shall be washed once with cold ether/alcohol and the Gooch and its contents dried in the oven on a weighed watch glass for two hours, the Gooch crucible for preference lying on its side.

The Gooch and its contents shall be cooled, weighed, and the percentage of wax in the sample calculated.

CLASS IX.—WAX

MELTING POINT.

I.P.T. Serial Designation—W 41.
A.S.T.M. Serial Designation—D 87—21 T.

DEFINITION.

1. The Paraffin Wax melting point is defined as the temperature at which melted paraffin wax, when allowed to cool under definite specified conditions, first shows a minimum rate of temperature change.

APPARATUS (FIG. 24).

2. *Wax Container.*—Test tube of standard form, 25 mm. (1 in.) outside diameter and 100 mm. (4 in.) long. It may be marked with a filling line, 2 in. above the bottom. This test tube shall be closed by a tightly fitting cork having two openings, one at the centre for the melting point thermometer and the other for a stirrer at one side of the centre. The opening for the stirrer may be lined with glass or metal tubing to act as a guide for the stirrer.

3. *Air-bath.*—Suitable water-tight cylinder, 2 in. inside diameter and 4½ in. deep. This air-bath shall be provided with a tightly fitting cork having a central opening for holding the test tube firmly in a vertical position in the centre of the air-bath.

4. *Water-bath.*—Suitable cylinder, 5⅛ in. inside diameter and 6 in. deep. This water bath shall be provided with a suitable cover and with the guides and fasteners necessary to hold the air-bath firmly in a vertical central position so that the sides and bottom of the air-bath shall be surrounded by a layer of water 1½ in. thick. The water-bath cover shall have a slot for introduction of a suitable stirrer and shall have an opening for the bath thermometer so that the latter may be suspended in a vertical position ¾ in. from the outside wall of the water-bath. Air-bath, water-bath and water-bath cover may be conveniently made of metal in one assembly as shown in Fig. 24.

5. *Stirrer in Test-tube.*—Brass or copper wire, ⅛ in. in diameter and about 12 in. long. A circular loop, ½ in. in diameter, shall be formed at one end of this wire in such a manner that the loop

lies in a horizontal plane when the rest of the wire is in a vertical position. The stirrer thus formed shall be passed through the proper opening in the test-tube cork and the upper end may then be bent into a shape convenient for holding.

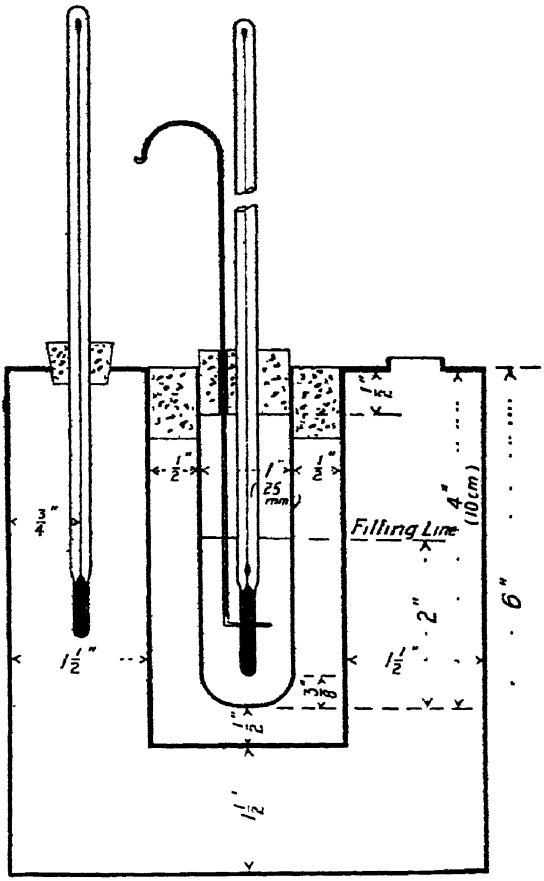


FIG. 24.

APPARATUS FOR DETERMINATION OF MELTING POINT OF PARAFFIN WAX.

6. *Thermometer.*—The I P.T. Paraffin Wax Melting Point thermometer shall conform to the following specifications :—

Type : Etched stem glass.
Total length : Approximately 370 mm.
Stem : Plain front, enamel back, suitable thermometer tubing. Diameter 6 to 7 mm.

Bulb : Powell's Normal, Jena 16 III or equally suitable thermometric glass. Length, maximum, 28 mm. Diameter not greater than stem.

Actuating liquid : Mercury.

Range : 80° to 160° F.

Immersion : 8 cm. The words " 8 cm. Immersion " shall be etched on the stem and also a line around the stem to indicate the depth of immersion.

Distance to 80° line from bottom of bulb : 105 to 115 mm.

Distance to 160° line from bottom of bulb : 330 to 345 mm.

Contraction chamber : Top to be not more than 41 mm. from bottom of bulb.

Expansion chamber : To contain mercury at 212° F.

Filled : Nitrogen gas.

Top finish : Plain.

Graduation : All lines, figures and letters to be clean cut and distinct. Scale graduated in 0°·2 F. and numbered every 2° F., every full degree line to be longer than the others.

Special markings : " I.P.T. Wax M. Pt." Serial No. and vendor's or maker's name or trade mark etched on the stem.

Each thermometer shall be tested at the National Physical Laboratory, where satisfactory thermometers will be marked with the N.P.L. monogram, and year of test, and a certificate of corrections issued.

7. Bath thermometer of any suitable type, accurate to 2° F. throughout the required range.

METHOD.

8. An average sample of the wax to be tested shall be melted in a suitable container in a water-bath whose temperature shall be not more than 35° F. above the approximate melting point of the wax sample. Direct heat, such as a flame or hot plate, shall not be used and the wax sample shall not be held in the melted condition any longer than necessary.

The test-tube shall be filled with melted wax to a height of 2 in. The test-tube cork, carrying the stirrer and the melting point thermometer with the 8 cm. immersion line at the under surface of the cork, shall be inserted into the test-tube for a distance of $\frac{1}{2}$ in. The lower end of the thermometer bulb shall then be $\frac{3}{8}$ in. from the bottom of the test-tube.

The air-bath being in its proper position in the water-bath, the latter shall be filled to within $\frac{1}{2}$ in. of the top with water at a temperature 15° to 20° F. below the approximate melting point of the wax sample.

The test-tube containing the melted wax, with wax stirrer and thermometer in place, shall be inserted into the air-bath in a central vertical position so that the bottom of the test-tube is $\frac{1}{2}$ in. from the bottom of the air-bath. The temperature of the water bath shall be adjusted by stirring if necessary, so that it shall be lower than the temperature of the wax sample by not

more than 30° F. and not less than 25° F., when the wax sample has cooled to a temperature 10° F. above its approximate melting point.

When these conditions have been obtained, temperature adjustment and stirring of the water-bath shall be discontinued. The wax shall be stirred continuously during the remainder of the test, the stirring loop being moved up and down throughout the entire length of the test-tube in a steady motion at the rate of 20 complete cycles per minute. The melting point thermometer reading, estimated to 0·1° F., shall be observed and recorded every 30 seconds. The temperature of the wax will fall gradually at first, will then become almost constant, and will then again fall gradually.

The melting point thermometer reading, estimated to 0°·1 F., shall be observed and recorded every 30 seconds, for at least three minutes after the temperature again begins to fall after remaining almost constant. The record of temperature readings shall then be inspected and the average of the first four readings that lie within a range of 0·2° F. shall be considered as the uncorrected melting point. This temperature shall be corrected if necessary for error in the thermometer scale and the corrected temperature shall be reported as the "I.P.T. Paraffin Wax Melting Point."

ACCURACY.

9. Duplicate determinations on the same sample should differ by not more than 0·2° F.

CLASS X.—CRUDE OIL

SPECIFIC GRAVITY.

I.P.T. Serial Designation—C.P. 1.

The apparatus and method must be chosen from the general directions for specific gravity according to the characteristics of the sample (p. 1).

DISTILLATION.

I.P.T. Serial Designation—C.P. 3.

APPARATUS.

The apparatus used shall be as laid down for the Distillation of Kerosene (K. 3, p. 17), except that the thermometer bulb shall not be covered with cotton-wool.

The graduated 160 c.c. measuring cylinder shall comply with the specification for the measuring cylinder as laid down under G. 3 (p. 7).

METHOD.

One hundred cubic centimetres of the oil measured at the laboratory temperature shall be distilled at a rate of 2–2½ c.c. per minute (approximately 1 drop per second), the distillate being collected in a clean dry receiver of the standard dimensions. The mouth of this receiver shall be covered with wet filter paper to minimise evaporation losses. The distillation shall be stopped at such a point that the temperature recorded reaches but does not exceed 150° C. The temperature shall be allowed to fall to 130° C., and the heating repeated in the same way several times until a further operation yields not more than 1 drop of distillate.

The total volume of distillate to 150° C. thus obtained and its specific gravity at 60° F. shall be recorded.

The distillation shall be continued in the same way, the distillate between 150° C. and 300° C. being collected in a clean dry receiver. The distillation shall be stopped at such a point that the thermometer reaches but does not exceed 300° C. The temperature shall be

allowed to fall to 280° C. and the heating repeated in the same way several times until a further operation yields not more than 1 drop of distillate.

The total of distillate between 150° C. and 300° C. thus obtained and its specific gravity at 60° F. shall be recorded.

The residue in the flask shall be allowed to cool to the laboratory temperature and transferred to a measuring cylinder of the standard dimensions, the flask being allowed to drain for five minutes. The volume of this residue and its specific gravity at 60° F. and the loss (100—vol. of distillates—vol. of residue) recorded.

The barometric pressure shall be recorded, but no corrections for pressure or for exposed thermometer stem are to be applied.

Larger quantities should be distilled in a suitable type of apparatus.

TOTAL SULPHUR.

I.P.T. Serial Designation—C.P. 4.

The method shall be as laid down for Gas Oils, G.O. 4 (p. 25).

FLASH POINT.

I.P.T. Serial Designation—C.P. 7.

For oils flashing below 120° F. the Abel Apparatus, K. 7 (p. 18) shall be used.

For oils flashing above 120° F. the Pensky-Martens Apparatus G.O. 7 (p. 28) shall be used.

VISCOSITY.

I.P.T. Serial Designation—C.P. 8.

The apparatus and method shall be as laid down under L.O. 8 (p. 45).

CARBON RESIDUE.

I.P.T. Serial Designation—C.P. 9.
A.S.T.M. Serial Designation—D 47—21.

The apparatus and method shall be as laid down under G.O. 9 (p. 36).

ASH.

I.P.T. Serial Designation—C.P. 10.

The method shall be as laid down under G.O. 10 (p. 38).

COLD TEST.

I.P.T. Serial Designation—C.P. 11.
A.S.T.M. Serial Designation—D 97—22 T.

The apparatus and method shall be as laid down under G.O. 11 (p. 38).

HARD ASPHALT.

I.P.T. Serial Designation—C.P. 12.

The method shall be as laid down under F.O. 12 (p. 69).

SOFT ASPHALT.

I.P.T. Serial Designation—C.P. 13.

METHOD.

Five grams of well mixed oil in a 300 c.c. glass stoppered bottle shall be dissolved in 135–140 c.c. of ethyl ether of sp. gr. 0·72 at room temperature. With constant shaking 70 c.c. of 96 per cent. alcohol shall be added slowly from a burette. After a final shaking the solution shall stand 5 hours at 15° C., then be filtered rapidly through a pleated filter.

The bottle and filter shall be washed with a mixture of 96 per cent. alcohol and ether (1 : 2) until about 20 c.c. of the filtrate evaporated give only traces of tarry matter. The washed asphaltic pitch, which still contains paraffin and soft resins, shall be dissolved from the sides of the bottle and from the filter paper by means of hot benzol and evaporated in a tared glass dish containing a weighed glass stirring rod. The residue shall be boiled with 30 c.c. portions of absolute alcohol, rubbing with a glass rod, until extracts on cooling and shaking give no precipitate of paraffin ; it shall then be dried at 100° C. and weighed.

I.P.T. Serial Designation C 87

The wax shall be determined in the whole of the distillate coming over above 300° C. from 100 grams of oil. After noting the weight of the distillate the wax shall be determined as follows :—

APPARATUS (FIG. 25).

Apparatus and Reagents.—Large test tube ; solvent consisting of equal parts by volume of absolute ethyl alcohol and anhydrous ethyl

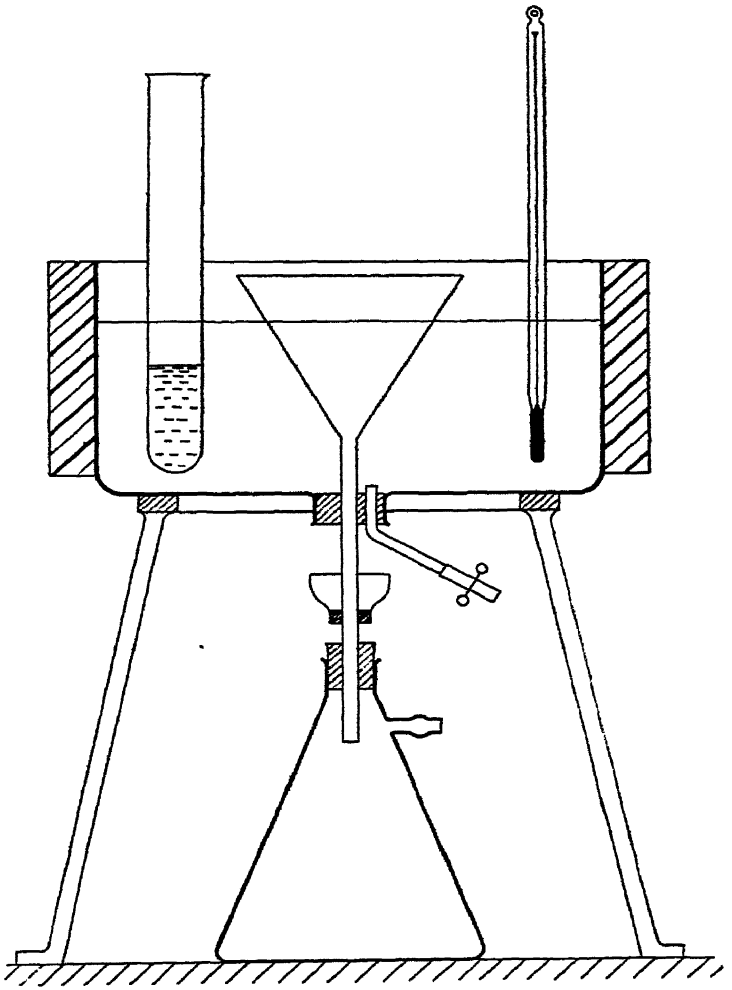


FIG. 25.

FILTER FOR WAX IN CRUDE OIL.

ether; apparatus of Fig. 23, which consists of a funnel so arranged in a bath that it may be surrounded by salt and ice mixture while filtration is going on; thermometer reading to –20° C. (–4° F.); Erlenmeyer flask; Liebig condenser; benzol.

METHOD.

Three to five grams of the sample shall be weighed into the test-tube and dissolved in the absolute alcohol-ether mixture, sufficient of the latter being added to give a clear solution. The solution shall be cooled in salt and ice mixture to a temperature of –20° C. (–4° F.), and sufficient of the absolute alcohol-ether mixture (cooled also to –20° C.) added to bring all oily drops into solution, leaving only flakes of paraffin visible. After cooling for 30 minutes, the precipitated paraffin shall be filtered with slight suction, the funnel being surrounded by a freezing-mixture of salt and ice.

The precipitate shall be washed well with cooled absolute alcohol-ether solution until free from oil, and then dissolved in hot benzol, which is run into a weighed small Erlenmeyer flask. The solvent shall be distilled off through a condenser, and the flask and its contents heated to 100° C. (212° F.) for 30 minutes in an oven. The flask and the wax which it contains shall be cooled in a desiccator, and then weighed. The percentage by weight of wax in the distillate above 300° C. (572° F.) is calculated, and from this the percentage by weight of paraffin wax in the original crude petroleum is found.

WATER AND SEDIMENT.

I.P.T. Serial Designation—C.P. 14, 14a.
A.S.T.M. Serial Designation—D 95—21 T, or
A.S.M.T. Serial Designation—D 96—21 T.

The distillation method described under F.O. 14 (p. 70) or the centrifuge method F.O. 14*a* (p. 73), shall be employed, according to whether water alone or water and sediment is required.

INDEX